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Development of Particle Size Test Methods for Sampling High Temperature and High Moisture Source Effluents

CALIFORNIA ENVIRONMENTAL PROTECTION AGENCY
AIR RESOURCES BOARD
Research Division

Development of Particle Size Test Methods for Sampling High Temperature and High Moisture Source Effluents

Final Report

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CONTENTS

LIST OF FIGURES

LIST OF FIGURES (cont'd)

SECTION 1

INTRODUCTION

The bulk of source testing for particle size distributions has been performed in flue gas streams downstream of heat recovery and pollution control devices which yield relatively low temperature, low moisture environments. These typically produce effluents with temperatures in the range of 250-500°F and less than 15% moisture. With the increasing use both of scrubbers for chemical pollution removal and incinerators for hazardous, industrial and municipal waste disposal, the necessity to sample high temperature and high moisture flue gases intensifies. In view of significant technical difficulties induced by these extreme conditions, no standard or generally acceptable methods of determining particle size distributions under these conditions have been developed and formalized. This poses a particular problem for regulatory agencies such as the State of California Air Resources Board (ARB), which must characterize and measure emissions for toxic substances under California's Toxic Air Contaminant Program.

In the last 15 years the state-of-the-art of particle sizing in industrial gas streams has matured substantially. Measurement of particle size distribution or size specific particulate emission rates has been driven by control device evaluation, environmental risk assessment, and regulatory applications. In the last decade formal emission measurement methods based on inertial particle sizing were promulgated, e.g., CARB Method 501 and EPA Methods 201 and 201a. These methods were developed by Southern Research Institute (SRI) using cascade impactors and cyclones.

The application of instrumental methods, principally optical devices, to particle size measurement in industrial process streams began in the early 1970's for special purpose applications requiring real-time monitoring. At that time available devices were built for ambient temperature and dry gas; so substantial modifications of the sample stream were necessary in order to use them. Since then several development efforts have been directed toward eliminating the necessity of sample extraction. Several devices incorporating optical sizing methods for use in industrial process streams have been offered commercially. Field experience is leading to improvements and at least one commercial probe has survived sampling in a combustion zone.

The conditions addressed by this project go beyond previous developments to include materials and handling problems associated specifically with high temperature and with familiar, but unsolved, problems of wet or saturated effluent streams. High temperature poses severe handling, validation, and corrosion problems using any available mechanism for providing particle size distribution data. High moisture with entrained or condensed water can overload sample collection substrates in inertial sizing devices before an adequate sample quantity (on a dry basis) is collected (or worse, entrained water can

wash the sample from the substrate). High moisture also increases errors associated with deposition of particles on extraneous surfaces of the sampler by eliminating re-entrainment without agglomeration. Entrained and condensed water has made frequent removal and cleaning of the windows of *in situ* optical probes necessary in many high moisture environments. Of course for environmental assessment purposes entrained water droplets and condensed moisture obscure the ultimate particle size of the dry solids at atmospheric moisture levels.

The specific applications of interest to the ARB, as expressed in the RFP for this work are:

High temperature-

- Uncontrolled hospital and municipal waste incinerators
- Flares at landfills
- Precontrolled effluent from cement kilns and incinerators for illicit drugs

and

High Moisture-

- Scrubbers used in pulp and paper manufacturing
- Steam generators for thermally enhanced oil recovery.

The emissions from flares at landfills, because they are essentially the result of burning combustible gases in air, are expected to have size distributions that are confined to the fine particle and submicron ranges of particle sizes. The remaining high temperature sources will tend to produce particles having a much broader range of sizes, extending from the submicron to at least several tens of microns. The primary particulate matter in the effluent from scrubbers in the pulp and paper industry is predominantly small while that from uncontrolled incinerators and cement kilns can be a high concentration mix of fine and coarse particles. The water droplets of concern in the effluents of the scrubbers tend to be relatively large scrubber-spray carryover, large mechanically entrained droplets from structural surfaces, and much smaller "fog" droplets that result from condensation in the gas stream as it cools. The sizes of the first two are of the order of several tens to hundreds of microns while those from the latter tend to be on the order of 10 microns. The droplets of concern from steam generators would be expected to have somewhat the same size characteristics of those from scrubbers; that is large mechanically formed drops and much smaller condensation fog droplets.

The objective of this project was to select and develop methods for successful particle sizing in both high temperature and high moisture flues. The basis for selecting the methods was a literature review of recent developments which might impact or contribute to proposed solutions of this problem, coupled with in-house expertise developed in conjunction with past and ongoing work for EPRI and the US EPA for measuring scrubber droplets and effluents and work for the EPA, EPRI, and DOE on measuring particle size distributions in high temperature gas streams. An interim report, "REVIEW OF PARTICLE SIZING METHODS AND EQUIPMENT" (November, 1992), provided to the ARB as part of this contract, contained a compilation of summaries of applicable findings from the literature search. This review was combined with the experience that SRI personnel have gathered in sampling high temperature and high moisture gas streams to arrive at a set of recommendations for the development of specific methods for the ARB applications.

There appeared to be two major approaches to high temperature or high moisture sampling: those based on the optical properties of particles and those based on inertial properties of particles. Each is discussed in this report in general categories. Some aspects of one or both of these techniques in the high temperature environment is suggested several possible solutions including: high temperature materials to construct samplers of existing designs, the cooling of in situ probes, and the use of dilution and cooling when extracting the sample gas. Approaches for the high moisture environment consisted primarily of a combination of using some type of inertial precollector to remove the large droplets and heating the remaining extracted gas so that sizing of the dried particles could be accomplished. Some additional techniques were also suggested for use in situations in which the sizes of droplets themselves are of interest. After consultation with, and obtaining the approval of, the ARB, one approach for each of the two source categories was selected for development for use by the ARB.

Defining the measurement condition of interest for the particles was no trivial or inconsequential matter in regard to the choice of technique to recommend or use. If the particles of concern are indeed those found in the stack at the process condition of high temperature, then the use of conventional devices made from high temperature materials or optical devices that determine the size of particles at flue gas conditions would be the favored methods. If concern for determining the size of particles that will make their way into the eternal environment and make contact with man is the driving force, then an extractive, cooling technique for high temperature streams and heating to evaporate water for wet streams become as important as *in situ* techniques, if not more so. Our understanding was that one of the primary goals of the ARB measurement program is to predict deposition rates and exposure downwind of sources. Consequently, methods which provide data more nearly at ambient conditions than at stack conditions were to be favored, all other things being equal.

In addition to defining the conditions of the particles of interest, identifying the size range of greatest importance also strongly influenced the methods recommended and selected. If the purpose were to document the general distribution of particle sizes being emitted, then perhaps none of the techniques would have been of greater benefit than another, or a combination of techniques may have been needed. If, however, there was a size range of primary concern, say of those sizes that are perhaps the most damaging to the human respiratory tract (the 0.2 to 10 μm range) (Trolinger and Bachalo, 1977; Spinosa et al., 1979; Spinosa and Holman, 1981; Pyle and Smith, 1984; McCain et al., 1986b) or some other target range of interest, then the choice of method would have to favor the technique(s) that best sampled and characterized that range. Discussions with personnel of the ARB and with outside consultants to the ARB led to the conclusion that the conditions of interest for characterizing the particles were those that would be found downwind from a source, with emphasis on the particles in the smaller size fractions.

SECTION 2

FUNDAMENTALS OF BASIC PARTICLE SIZING TECHNIQUES

2.1 Introduction

In the literature concerning procedures for particle size measurements, one finds methods to be generally divided into non-extractive and extractive techniques. Most of the non-extractive techniques use some form of optical methods to record, measure, or estimate the particle size. The most common of the extractive techniques involve some aspect of the inertial, electrical, or diffusional properties of the particles, although there are some optical devices that require extraction. Of the extractive methods, the ones that will receive primary focus in this report are those that depend on the inertial properties.

Before proceeding with descriptions of the methods selected, summaries of the underlying physical principles of various potential techniques will be presented together with some discussion of their relative advantages and disadvantages.

2.2 Optical Methods

The desirability of optical devices for data acquisition stems from their potential as sensitive, rapid, real-time, in situ, and non-intrusive process monitors (Pyle and Smith, 1984). Coleman (1977) looked into systems for measuring particle size distributions in high temperature, high pressure flows resulting from advanced, open-cycle gas turbine systems fired with pulverized coal and containing polydisperse systems of particles of unknown shape, index of refraction and composition. He concluded that there were, at that time, no commercially available instruments for reliable measurement of particle sizes under these conditions. Imaging (photography and holography) and scattered light (visibility, diffraction, and intensity ratio) techniques were identified by Coleman as the most promising for further development. The focus of his review was the on-line, near real-time identification of particles capable of damaging turbine blades. This premise led to different assumptions and criteria from those of this study; therefore, not all of his conclusions are directly applicable.

Light scattering methods can be broken into two major categories - those based on the measurement of individual particles and those based on the measurement of the properties of light scattered by a large ensemble of particles.

2.2.1 Underlying Physical Principles

2.2.1.1 Imaging systems

Of the imaging methods, photography is a fairly standard technique using either a spark or pulsed laser beam to illuminate the system of interest. The optical system can be designed to magnify the particle images if necessary before transmission to the recording device, either a photographic emulsion or a video camera. Either direct imaging, as in conventional photography, or an indirect imaging technique, holography, may be used. Holography can be used to provide a basis for recording not only the physical appearance of the particles but their spatial relationships as well. A hologram is formed by splitting a laser beam into a beam which illuminates the object and a reference beam which is reflected with phase unchanged. Both beams fall on the holographic emulsion, with the phase difference between them (caused by the object) forming an interference pattern on the emulsion. The image is reconstructed by illuminating the hologram with a coherent light source. A wave pattern formed behind the hologram appears as images of the original objects.

Coleman (1977) emphasized optical techniques in his study because they have the potential to provide non-perturbing, in situ measurements. Specific to the imaging techniques, holographic methods potentially contain the most information (Hirleman, 1982). It is generally agreed, however, that the lower limit on resolution of photography techniques using visible light is in the 2-5 μm range. Holography has been used to measure particles sizes down to the 5-10 μm diameter range (Coleman, 1977; Hirleman, 1982; Hsu et al., 1991). However, holographic methods require very sophisticated and expensive processing (Hirleman, 1982) and the optical layout can be difficult to place and use for flows in large ducts. Holographic methods were not found to be completely satisfactory for on-line monitoring of the particles within gasifier exhaust ducts by Hemsley et al. (1985) because of difficulties in signal collection and processing.

With regard to direct imaging methods, SRI developed a video based imaging system for in situ measurements of the size distribution of spray droplets and entrained water in scrubbers (McCain, 1989). The latter system provides data over a 150:1 range of particle sizes selected by the user within the overall range of 3 μm to 6000 μm (i.e., 3-450 μm, 20-3000 μm, etc.). Although designed for use in the relatively low temperature environments of scrubbers (<200°F), with additional cooling such a system could be used at higher temperatures. One major drawback of this and any of the optical methods is a lack of discrimination between solids and liquids.

2.2.1.2 Non-imaging (or light scattering) systems

The measurement of particle size distributions by means of light scattering properties can be done either by illuminating one particle at a time or on the basis of the integrated light scattered from an illuminated ensemble of particles. The former offers the promise

of high resolution with respect to particle size at the expense of requiring rather low particle concentrations; while the latter tends to require high particle concentrations and provide results in the form of distribution functions characterized by two or three parameters without the capability of resolving fine detail in the distributions.

Single particle methods -

Several techniques have been used for making in situ measurements of individual particles. One of these is the fringe visibility method in which particles traverse a sample volume defined by the intersection of two coherent laser beams. Interference of the two coherent beams forms a fringe pattern in the illuminated measurement volume. Modulated signals result as individual particles move through the fringe pattern. The size of a particle is deduced from the percentage of modulation in the amplitude of the scattered light signal. (The modulation frequency is used in "Laser Doppler Velocimeters" for measuring gas velocities in particle laden streams.)

Other methods based on light scattering by individual particles rely on the intensities of light scattered over a selected set of angles or the ratios of the intensities of light scattered at different angles from the forward direction to determine particle size. A beam of light is focused within the flow system, and the scattered light is collected at desired angles from the forward direction by use of apertures and light pipes, lens and mirror arrangements, or an array of photodetectors.

Farmer et al. (1983) reported results of particle size and velocity measurements in a 4580°F (2800°K) combustor using a Particle-Sizing Interferometer (PSI), a device using the visibility technique. It was set to give an effective particle-size range of 1.8-38.4 μm. Hemsley et al. (1985) found that since the "Doppler" (modulated) signal visibility is a ratio of two parameters which are equally affected by window fouling and laser beam attenuation by other particles in the beams before the probe volume, it was considered to be a more reliable function of particle size in hostile environments than that achieved by monitoring diffracted intensity alone. The visibility technique is dependent on having only one particle in the measuring volume at any one time (Coleman, 1977). This translates to maximum particle loading densities of the order of 10^4-10^5 particles/cm^3 (Coleman, 1977; Hsu et al., 1991). The number densities of particles larger than 1 to 2 μm in uncontrolled sources is typically on the order of 10^6 particles/cm^3, making the potential use of such devices questionable for such applications. Moreover, where small particles (< 0.1-0.2 μm) are present in addition to larger particles (> 1-2 μm), signals from the smaller particles in the measuring volume will contribute to the background noise when compared with the Doppler signals from the large particles. Farmer et al. (1983) estimated the small particle content by making optical transmission measurements. These showed that a significantly large number of particles less than 0.1 μm in diameter were present in the combustor. As configured, the PSI was incapable of detecting particles in this size range. Experience in applying the visibility technique in industrial environments has indicated that on-site

calibration is needed to prevent systematic errors in the particle size data. Hsu et al. (1991) state that single-particle-counting techniques are generally limited to particle sizes larger than 0.5 μm.

Pyle and Smith (1984) discuss the Fine Particle Stack Spectrometer System (FPSSS) manufactured by Particle Measuring Systems, Inc. It uses single particle near forward light scattering to measure particles ranging from 0.4 to 11 μm. The probe uses a recirculating liquid coolant to keep the temperatures of the optical and electronic components at tolerable levels; but even with that, it is limited to gas temperatures below 500°F. According to Pyle and Smith (1984), the aerosol concentration at which coincidence of particles in the view of the detectors becomes significant is $5*10^4$ particles/cm^3 for the FPSSS. The effect of coincidence errors is greatest for small particles, so that the lower limit of sizing capability is raised as the concentration increases above this value. As the size of the sample volume is decreased, statistical effects at low concentrations become significant. Hemsley et al. (1985) claim that the use of single beam forward scattering instruments is not completely satisfactory for on-line monitoring of the particles within gasifier exhaust ducts because of difficulties in signal collection and processing.

Holve et al. (1988) and Bonin and Queiroz (1991) report that particle velocity, size, and number density measurements were made in full-scale, coal-burning power plants using the Particle-Counter-Sizer-Velocimeter Probe (PCSV-P) (a small-angle, forward-scattering device) developed by the Insitec Corporation. They claim it is the only technique currently available for measuring local number densities and size distributions for non-spherical particles. The practical size range spanned for this particular instrument is 0.5 and 80 μm at particle velocities as high as 400 m/s. Bonin and Queiroz (1991) point out that the PCSV-P is a single particle counter, meaning that the volume of the measurement sensing zone must be restricted. Coincidence problems are obviated to a great extent without sacrificing the counting statistics for larger, generally less numerous particles by sequentially using two different beams and scattering geometries in one probe. One beam with a very small sensing zone is used for measuring the generally more numerous small particles (0.5 to 3 μm) and another beam with a larger sensing zone is used for the larger particles (3 to 80 μm). Comparative data taken with the PCSV-P and cascade impactors at a coal fired power plant showed reasonable agreement for sizes between 0.5 μm and 5 μ. However, concentrations derived from the PCSV-P data were biased high by factors ranging from 10-fold to 100-fold for sizes above 10 μm as compared to the gravimetric determinations. Cenospheres (physically large, hollow balloon-like particles) were advanced as one possible cause of the discrepancies at the larger sizes. The diameters accorded them optically would be much larger than their aerodynamic diameters; and, because they are hollow, the masses assigned to them based on their physical diameters and assuming they were solid would be far too large. Errors in the sizes assigned to particles because of differences in assumed and real refractive indices may also have played a role, especially above 15 μm (see Section 2.2.2). When making measurements in high temperature or otherwise extreme environments, the PCSV-P optical system is inserted in a 10-cm-diameter, water-cooled jacket that can be extended up to three meters into the processing unit. This jacket is

designed to permit operation of the probe in gas temperatures up to 2600°F (1700°K). SRI experience with the probe indicates that window fouling makes it virtually unusable in streams containing entrained water and that the coincidence effects in flue gas streams may result in a gap in the measured size spectrum between about 3 and 12 μm when measuring uncontrolled emissions.

Particle ensemble methods -

Ratioing of the light scattered at two angles eliminates uncertainties due to variations in incident light intensity (Trolinger and Bachalo, 1977). Particles in the diameter range of 0.1-1.0 μm have been measured using the ratios of forward and back-scattered intensities. Wittig et al. (1989) refer to this method as the dispersion quotient technique. They made measurements at high particle concentration (above 10^7 particles/cm^3) in the size range from 0.1 to 0.6 μm. The operating conditions of their experiments were at relatively low temperatures (<170°F) and moderate relative humidities (0-40%). Comparison of the results obtained with their technique and those based on extractive sampling methods showed excellent agreement in terms of volume concentrations. Their measurements were highly reproducible. An ensemble light scattering technique, based on measurement of the polarization ratio, was used by Hsu et al. (1991) to measure fume particle size and number density from a synthetic smelt. This in situ non-intrusive technique was used because of its potential for application to streams with high number densities of particles. They claim that such light scattering ratio techniques have been used to determine particle mean size in the range of 0.01 to 200 μm with particle concentrations up to 10^{12} particles/cm^3. However, this technique determines particle size and number density based on an assumed particle size distribution function. They estimated such a function by collecting particles and using a scanning electron microscope (SEM) and an image analyzer. Thus the device did not itself provide particle size data.

Another technique based on diffraction determines the size distribution of particles within the cylindrical volume formed by a laser beam passing through a flow system from the angular distribution of the light in the Fraunhofer diffraction pattern resulting from an ensemble of particles within a cylindrical volume. The size distribution can be calculated by matrix inversion or a least squares determination of parameters in an assumed size distribution law.

Montagna et al. (1977) and VanValkenburg and Frock (1977) reported on the use of a multi-particle analyzer, the Microtrac developed by Leeds and Northrup Company, that uses the diffraction technique. The mean diameter and variance of the distribution for particles in the 1-50 μm range could be determined. They report good agreement between light scattering and cascade impactor methods for particle sizing, especially for high-loading measurements in a flue gas at 1500-2000°F. Montagna et al. (1977) note that, if the distribution of the particles is multimodal, it cannot be characterized with the Microtrac. Moreover, for low loading conditions, the mean diameters obtained were larger than mean

diameters obtained with the impactor (or gravimetric) methods. This may be explained by the fact that, for their low load tests, the loading measurement signal was only 4% greater than the background coupled with the fact that the Microtrac has a highly attenuated response to submicron size particles (VanValkenburg and Frock, 1977). The loading data as functions of time also show a slow oscillatory characteristic, indicating that, while its means of measuring particle size is independent of laser power, the indicated loading is directly proportional to laser beam intensity. Another potential limitation that VanValkenburg and Frock (1977) note is that for installations on larger ducts (Dia. >10 in.), the scattered flux from some particles 1-3 μm in size will be vignetted due to the limited size of the collecting optics. Very small particles at long distance from the collector lens scatter flux outside the lens which results in a bias against small particles in the measured size distribution. The Malvern Instruments particle sizing device operates in a similar fashion to the Microtrac and suffers from the same vignetting problem. Both require transmitters and receivers mounted opposite one another across the duct with the instrument components mounted external to the duct.

An in-stack Fraunhofer diffraction system also is marketed by Insitec Measurement Systems which can cover a span of about two decades in particle size. This system, the EPCS-P, uses a relatively short optical path (~4 cm); consequently it is limited to use in streams with moderate to high particulate concentrations. It can be constructed to cover a 100:1 range of diameter selected by the buyer in the interval from 1 μm to 500 μm (i.e., 1-100 μm, 3-300 μm, etc.). A water cooled jacket permits the device to be used at temperatures up to 2550°F. The manufacturer does not recommend using it for relatively clean streams such as those downstream of efficient particulate control devices.

Hybrid methods -

Some instruments use more than one measurement technique. The Particle Sizing Interferometer (PSI) manufactured by Spectron Development Laboratories, Inc. (Montagna et al., 1977; Trolinger and Bachalo, 1977; Pyle and Smith, 1984) is an instrument using two techniques that has been applied in high temperature environments (1000-2000°F). This PSI sizes particles over the range of 0.5 to 25 μm diameter. The intensity ratio optical method is used for particles with diameters of 0.5 to 3 μm and the fringe visibility technique is used to size particles with diameters 3 to 25 μm. This visibility technique is the same approach as the PSI of Farmer et al. (1983) discussed earlier.

Wertheimer et al. (1979) reported on the Optical Stack Particulate monitor manufactured by Leeds and Northrup, an ensemble light scattering device which measures concentrations in five size fractions over the size range from 0.1 to 10 μm. It uses the polarization dependence of light scattered at 90° to the illumination direction to measure the concentration in two submicron size bands and the scattered intensities at three angles in the near forward direction to deduce particle sizes and concentration rates over the range from 1 to 10 μm. Spherically-shaped particles are assumed for all the modeling of the instrument response.

2.2.2 Relative Advantages and Disadvantages

One of the principal advantages of at least some of the optical devices is that they are capable of providing size resolution to substantially larger sizes than can be provided by their major competitor: inertial methods which will be discussed later in this report). However, there are always sizes above and below which any of the optical devices do not register particles at all. Further, liquid and solid particles cannot be distinguished and even in the best of circumstances transformations from measured distributions on a concentration by number basis to concentration by mass can be fraught with error (as in the case of cenospheres).

Coleman (1977) concludes that because some of the primary particles in the exit flow of many combustion processes will be porous or hollow and agglomerates containing voids, there is no reliable method for converting optically-determined number density and size distribution measurements into mass loading density values. Despite considerable progress in laser/optical diagnostic techniques, Hirleman (1982) and Self (1982) raised a number of serious questions and uncertainties that remain concerning in situ laser light scattering measurements in coal combustion or other environments. The following non-ideal factors must be considered in all applications of optical methods for in situ analysis of particles in coal combustion environments: effects of particle shape, effects of unknown refractive index that may vary from particle to particle (Is the instrument calibrated with particles of one particular refractive index?), interference from particles outside the nominal sizing range (smaller or larger), size-dependent sampling bias or particle counting errors (sample all sizes with equal probability or have a calibrated means for correcting the data), and a requirement for field calibration to verify correct alignment and operation.

Figures 1 and 2 illustrate the effect of particle refractive index on the response of light scattering instruments. These show that under extreme conditions with unknown particulate refractive indices one can find sizing errors of greater than a factor of two and consequent mass errors of more than a factor of eight. The refractive index of particulate matter in industrial emissions is quite variable, depending on the exact composition of the particulate matter and may not be homogeneous with respect to size, even for a single gas stream. The range that is possible runs from 1.33 for water droplets to 1.95 -0.66i for materials like coal to 1.33 -0.1i for water droplets containing a moderately absorbing contaminant. The curves shown in Figures 1 and 2 relate to spherical particles and non-spherical particles add a further confounding factor. Self (1982) and Pyle and Smith (1984) also point out that these instruments may have other problems, such as dust build-up on the optical components, non-representative aerosol size or concentration at the sampling point, or corrosive effects of the process gas on delicate or sensitive components. The Fraunhofer diffraction devices are particularly susceptible to problems from fouling of windows as particles deposited on the windows contribute directly to the signal and are included as part of the measured aerosol. The optical instruments as a class also cover more or less limited ranges of particle size. None perform well, if at all, in measuring submicron particles and many are limited to relatively small upper limits to their detection

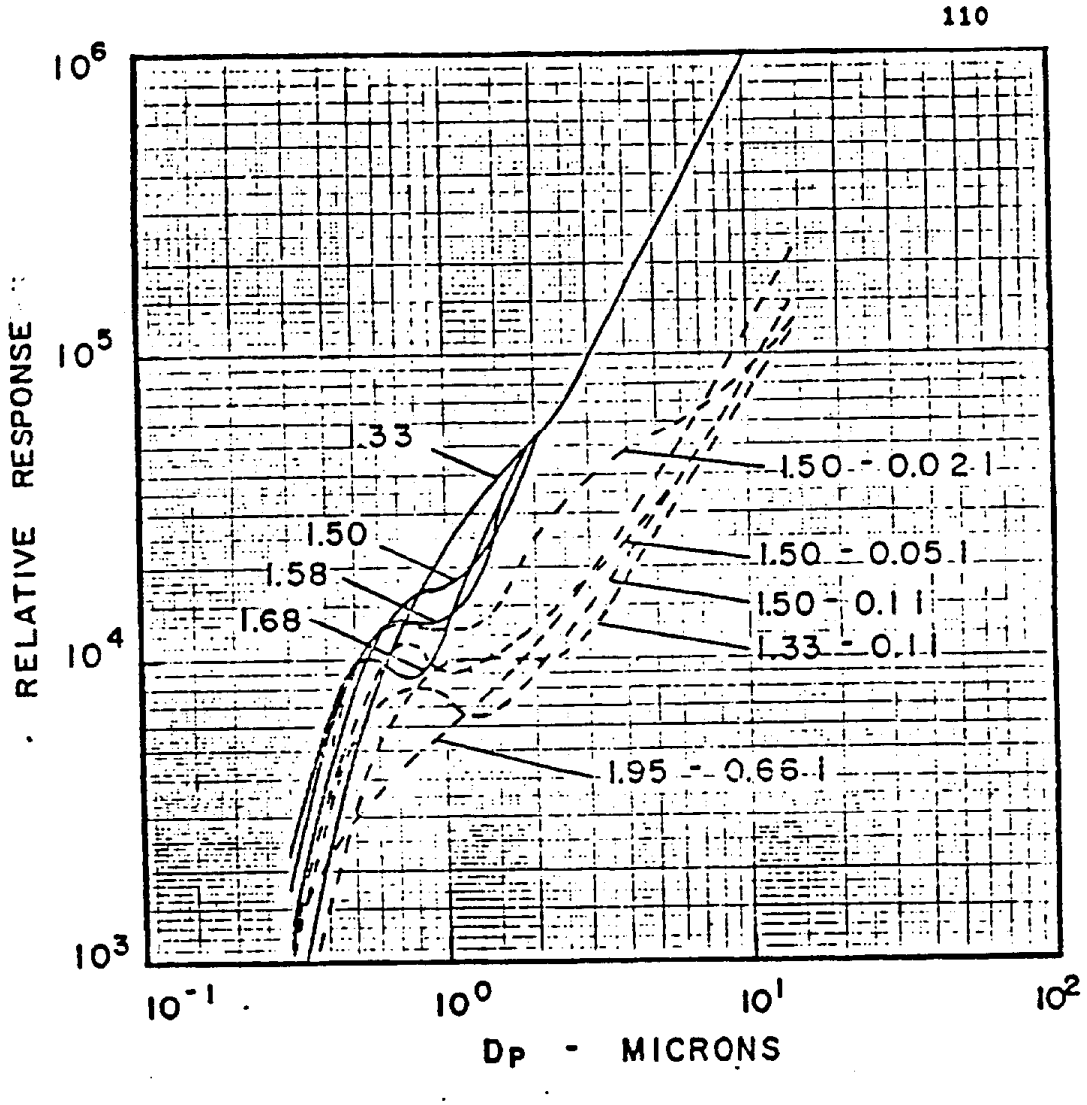
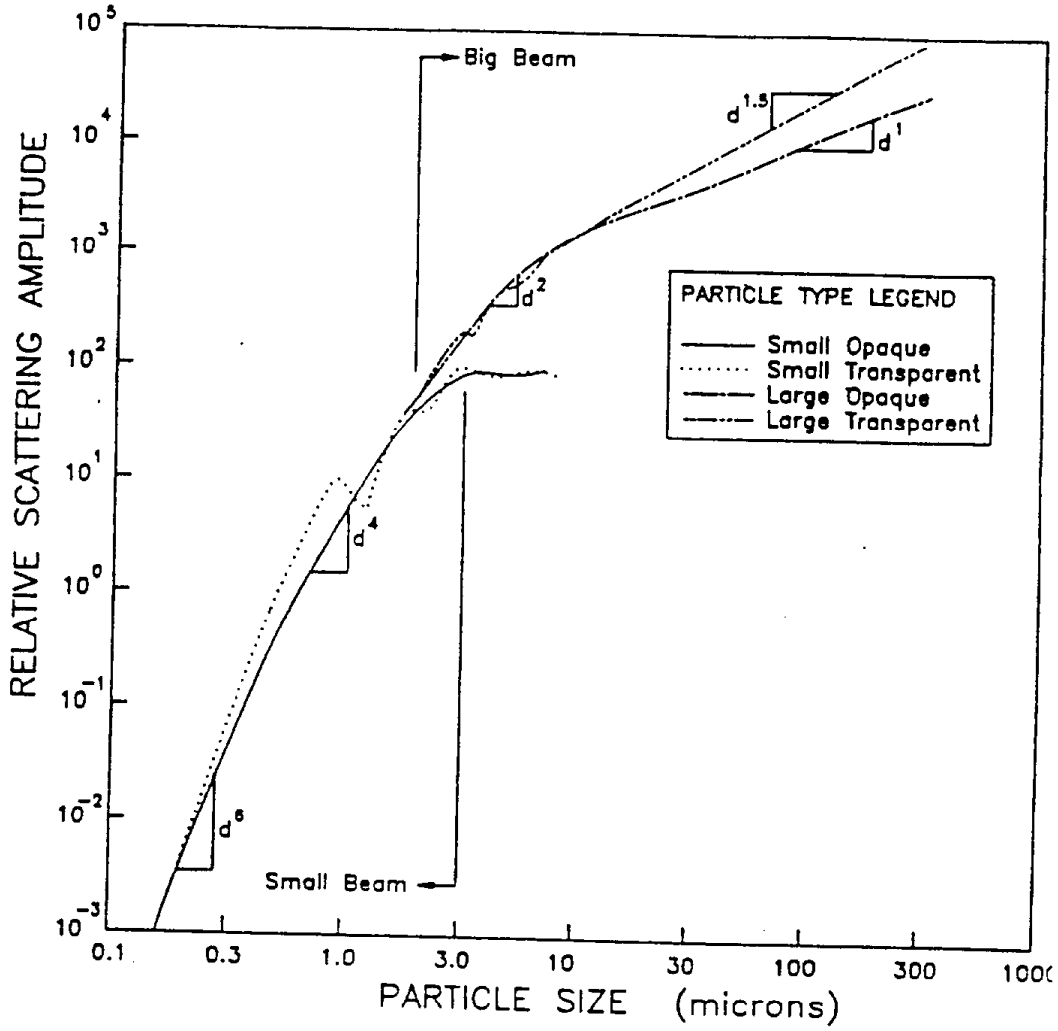


Figure 1. Relative response versus particle diameter for the Bausch & Lomb 40-1 optical particle counter for a range of particle refractive indices. (Berglund, 1972)



Note : Slope triangles show approximate power law dependence as a function of particle size.

Figure 2. Relative response versus particle size for the Insitec PCSV-P optical particle sizing probe for transparent particles with a refractive index of 1.6 and for absorbing (black) particles. Two optical beam geometries are included in a single probe. (PCSV-P Operator's Manual, 1991)

capabilities in terms of particle size and concentration.

In addition, optical instruments require sources of filtered compressed air for purging windows and must be cooled to maintain the temperatures of electrical and optical components at ambient conditions. Otherwise catastrophic failure will usually occur.

Single particle counters require moderately complex optical systems and signal processing electronics (Hirleman, 1982) which results in relatively high costs. The FPSSS has a purchase price of about $40,000 and the PCSV-P costs about $120,000. Construction of an SRI Video analyzer would cost about $150,000 for the low temperature version currently in use.

2.3 Inertial Methods

Two inertial techniques (cascade impactors and series cyclones) have been adapted for use in the environment of most industrial flues (McCain et al., 1986b). The majority of the particle-size data available on particulate matter suspended in industrial process streams, especially that emitted to the atmosphere, has been taken using cascade impactors (Pyle and Smith, 1984; McCain et al., 1986a). For measurements at relatively low particulate concentrations the cascade impaction is the preferred inertial sizing device. When measurements of particle concentration and size distribution are carried out at high particle concentrations or when a larger sample of size-fractionated particulate matter is desired, the use of series-cyclone sampling systems may be preferred. The current ARB method for measuring the particle size distributions of emissions from stationary sources, CARB Method 501, is based on the use of cascade impactors.

2.3.1 Underlying Physical Principles

The operating principle of a single impactor stage is discussed and illustrated Pyle and Smith (1984) and McCain et al. (1986a,b). Basically the device works by pulling flue gas into the sampler at a constant flow rate and then subjecting this sample gas stream to an abrupt change of direction. A round or rectangular orifice at 90° to a flat surface in close proximity to it is used to produce the change in direction. A suitable impaction surface, or substrate, is place on the flat surface opposite the orifice such that the inertia of any given particle determines whether it will follow the gas stream or impact on the substrate. Particles which do impact must be retained for the method to work. This leads to restrictions regarding the properties of the impaction surfaces and the geometries and flow rates used in the devices. However, the latter are well understood and practical designs are possible.

To measure a particle size distribution, the gas stream is made to sequentially pass through several such stages, each one forcing a more abrupt change in direction than the previous, thus larger particles impact in the upper stages and smaller particles impact in the

lower stages. A filter located behind the last impaction surface removes any remaining particulate matter. Analysis involves determining the size cuts produced by the various impaction stages and the weight gains for their respective substrates collecting particles in the size range(s) of interest. The amount of gas sampled (sample volume) must be sufficient to obtain measurable weight changes for all the substrates. The data thus obtained represents the average size distribution for the sampling period (McCain et al., 1986a). Sizing particles in this fashion has a sound theoretical and experimental basis, including that needed for predicting the size cuts produced based solely on the gas composition, impactor stage geometry, and sampling flow rate over a wide range of conditions-including the high temperature conditions of interest here (McCain et al., 1986a and Parker et al., 1981).

Cyclones are forms of centrifugal particle collectors in which the circulation of a particle laden gas stream about a central axis is induced by conversion of forward motion through the cyclone inlet. The gas enters through the inlet tube from whence it passes into the cylindrical body of the device. Here it acquires a spiral motion, descending along an outer spiral toward the base of the cyclone for some distance; it then undergoes an abrupt change of direction and continues upward to and through the exit tube. The flow in the central core of the cyclone, after the gas has turned upward, may proceed in a tighter inner spiral, or may flow in a rectilinear path to the outlet tube. The flow field within a cyclone is highly complex and cannot be modeled in two dimensions as has been done for impactors. Unfortunately, cyclones do not have an adequate theoretical relationship with which to predict their performance under field conditions solely from gas composition, cyclone geometry, and sampling flow rate. Typically theories to explain their performance are based on the classical equations for centripetal force and include additional terms to account for viscous drag on the particles, turbulence, and particle exchange between the outer and inner vortices. Early theories of cyclone operation included only the inertial forces due to the centripetal accelerations of the particles. Later developments included some of the effects of gas turbulence inside the cyclone body. One relation that has been used to predict the cyclone cut-point expresses it as a square root function of the cyclone inlet geometry, gas viscosity, particle density, volumetric gas flow-rate, and the effective number of turns made by the gas stream in the cyclone. A difficulty in applying such a relation is determining the effective number of turns of the gas stream as it travels through the cyclone, the value of which cannot be calculated a priori. Other theoretical relations for cyclonic particle collection have been proposed; however, none have proved adequate. Pyle and Smith (1984) describe, illustrate, and reference the problem more thoroughly. Basically, series cyclone operation is similar to that of an impactor in that the inertia of the individual particle is used to separate it from the sample gas stream, but this separation is caused by cyclonic action rather than impaction. The models usually attempt to relate the cyclone efficiency to cyclone dimensions, flow rate and aerosol properties. McCain et al. (1986b) more fully continue this description with some of the equations used and descriptions of some of the more current empirical models. Empirical relations for predicting the cut diameters of a specific set of commonly used sampling cyclones were developed by McCain et al. (1986b). However, the range of conditions covered in that

work did not extend to those appropriate for the operating temperatures contemplated here. Hence a significant amount of additional calibration effort would be needed in order to use new or existing cyclones with confidence at the high temperatures of interest here.

2.3.2 Relative Advantages and Disadvantages

Established inertial particle sizing methods rely on the extraction of a sample of the particulate matter by an isokinetic probe and off-line particle analysis. The cascade impactor is the most widely used in situ sizing method in process or ambient air in the size range 0.3-20 μm. It has several advantages over other particle sizing devices: many stages of size classification in a small volume; classification of particle size based on inertial and aerodynamic properties; its performance is well characterized for moderate temperature, ambient pressure operation; calculated variations in cut size with temperature have been experimentally verified for temperatures up to 800°C (1500°F); it is capable of being inserted directly into gas ducts; it provides reasonable accuracy; is rugged; is easily portable; and is adaptable to a large variety of aerosol streams (Montagna et al., 1977; Masters et al., 1979; Pyle and Smith, 1984; McCain et al., 1986a,b). Spinosa et al. (1979) and Spinosa and Holman (1981) reported on the determination of emissions from glass manufacturing operations using cascade impactors. Most of these 13 stacks ranged from 316 to 700°F, but one had a temperature in excess of 1600°F. (Unfortunately, they did not give details of the hot stack operations or of problems encountered, if any.) Conventional in-stack impactors can be used to size particles larger than about 0.3 μm aerodynamic diameter. Size classification of particles below 0.3 μm is possible by using very small jet holes (micro-orifice impactors) or by operating impactors at reduced gas pressures (low pressure impactors). Kauppinen and Hillamo (1989) reported on a modification of the University of Washington Mark 5 in-stack impactor design and operation to have equally spaced aerodynamic cuts on a logarithmic basis over the size range 0.03-15 μm.

Hirleman (1982) and Farmer et al. (1983) suggest that measurement probes such as cascade impactors are particularly difficult to use in measurements involving high-temperature flows. They concluded that either such devices introduce unwanted flow perturbations, are too slow, cumbersome, and inaccurate, or cannot survive the high temperatures. However, the flow perturbation problem mentioned by them was due primarily to the small duct sizes (<12 in.) encountered in their work. In general applications the duct dimensions will be larger and blockage of flow in the duct by the sampler should have little effect. Their other comments tend to hold for virtually any device other than that concerning speed. The inertial methods are definitely slow as compared to on-line real-time methods.

Montagna et al. (1977) used a cascade impactor, a Coulter counter, and absolute filters to determine particle size distributions and mass loadings in 1650°F flue gas from various locations in a pressurized fluidized-bed combustion system. They extracted the sample gas stream through an electrically heated sample line to maintain the temperature

of the gas sample above its water dew point. Because the Coulter counter was calibrated with standard particles and its measurements of combustion particles agreed well with cascade impactor measurements, it was assumed that these comparative measurements were representative of the true particle distributions. These comparisons were carried out to provide reference data for evaluating the PSI and Microtrac optical devices that were described previously.

Masters et al. (1979) reported successful development and demonstration of a system using an extractive approach, removing samples from the process stream for complete analysis of particle size distribution. The sample was withdrawn isokinetically from the 1350°F stream. A cascade impactor (Mark III, University of Washington Source Test Cascade Impactor, Model D) was used to collect and size particles at 450°F. This temperature was selected with the awareness that possible changes in particle composition would have to be considered. They concluded that under their sampling conditions major changes in composition are not likely above the sulfuric acid dew point although possible changes of trace element concentrations might be of some concern. The probe and nozzle arrangement used by Masters et al. was similar to that used in Method 5. This work was done before the effects of particle impaction in a nozzle on size distributions measurements were recognized as being serious. Hence, they did not consider the modifications to the measured size distribution that would result from the use of conventional method 5 type 90° nozzles.

A similar technique to that of Masters et al., sample extraction and cooling to 500°F, has been used by SRI in measuring the size distributions of particles in the gas streams of a pressurized fluidized bed combustor and a coal gasifier with temperatures in the range of 1400°F to 1500°F. In the latter cases steam traced probes approximately five feet long were used to achieve the desired sample gas temperatures at the impactor inlet and the impactors themselves were operated in ovens. Straight vertical probes with straight integral nozzles were used to sample from ducts in which the flow was vertically down. This arrangement eliminated losses in bends and minimized transport losses in the probes. High operating pressures (10 to 25 atm.) required that the probes be permanently emplaced, precluding the use of a removable in duct precollector for handling the larger particles. This arrangement can be used only in special circumstances when the duct geometry is suitable (which will usually not be the case). Coleman (1977) and Self (1982) warn that dilution and/or cooling of the sample may induce substantial composition and particle size changes due to agglomeration and condensation of gaseous species, in particular, water.

Cascade impactors in their current commercial form are labor intensive devices which yield data representing time averages for a sample period that may range from a few minutes in high concentration flows to a few hours (or even days) in low concentrations (Trolinger and Bachalo, 1977; McCain et al., 1986b). Moreover, they require a coating or other substrate to minimize the problem of particle bounce. Both grease and fibrous impaction surfaces are subject to chemical and/or physical changes when exposed to

industrial flue gases. These effects will tend to become more serious at higher temperatures, up to the point of complete breakdown. As they deteriorate, the adhesives or surface coatings used for particle retention in impactors can result in intolerably high backgrounds and/or interferences. The quantity of particulate matter which can be collected on a single stage before overloading or re-entrainment occurs (a few milligrams at most) may place undesireably short limitations on sampling time. The shorter the sampling time, the greater may be the uncertainty in obtaining a representative sample, especially in a variable flow. Particle size dependent effects in the sampling nozzles and inlet transforms used to withdraw the sample from the gas stream to be measured and deliver it to the impactor stages must also be accounted for in the measurement process. Losses in bends, expansion zones, interconnecting tubing, and housings can arise from inertial deposition, turbulent deposition, and gravitational settling. Models for estimating losses from these mechanisms have been developed recently by Anand et al. (1992), McFarland et al. (1991) and McCain and Hoover(1992).

Cyclones have the distinct advantage that they can collect large quantities of samples (up to several grams) in each cyclone in the set without overloading (Pyle and Smith, 1984; McCain et al., 1986b). Therefore, they may be used to good advantage for measuring particle size distributions when the particulate concentration in the stream to be sampled is very high. Moreover, cyclones do not suffer problems from particle bounce or retention and require no surface coatings which might lead to interferences. Masters et al. (1979) used a titanium in-stack scalping cyclone because they found it readily available, small enough for insertion into the duct, and very efficient in its 0.6-μm cut-point. During the tests at 1350°F conditions, however, the cyclone's protective gold plating blistered and fell off, leaving the titanium surfaces exposed to heavy oxidation. Wang and Libkind (1982) reported on the development and use of a particulate mass analyzer incorporating a set of cyclones, each with its own tapered-element, oscillating microbalance (TEOM). The sampling probe and cyclone train were heated electrically to the process-stream conditions (1520°F) to keep the sampled gas from cooling. The cyclones were a set modified from an Acurex Source Assessment Sampling System (SASS). They were redesigned with flanges and thicker material for high-temperature, high-pressure operation. The cyclone trains had particle cut-sizes of 10, 3, and 1 μm at room temperature. Because of the temperatures encountered, the LED and photo-transistors used to monitor the oscillation of the fiber mass sensing elements were placed in a box outside the detector assembly. Two high-temperature optical fibers were used to transmit the LED signal and to carry the oscillation signal to the photo-transistor. Although the sampling probe and cyclones were designed to be kept at the process-stream temperature, the TEOMs were kept below 440°F by insulating flanges between the TEOM housing and the cyclone. With this system, real-time particle mass-loading and size-fraction measurements were obtained for the first time downstream from hot-gas cleanup equipment during a continuous four hour run.

One drawback to cyclones is the fact that particulate catches are frequently distributed over rather large surface areas within the cyclone. This can make sample recovery difficult if the particulate concentration is low unless very long sampling times are

used. On the other hand, overloading of stages when sampling high concentration gas streams is not a problem with cyclones and the large samples typically obtained when sampling high concentration streams make sample recovery easy. Because current theories on cyclone operation do not well predict cyclone performance from geometrical and flow considerations, extensive calibrations are needed so that good empirical relationships can be developed for use in calculating the sizes of the collected particles (McCain et al., 1986b). Extrapolation of cyclone performance to high temperatures based on currently available calibration data is not on nearly as sound a basis as is the case for impactors. Thus significant additional calibration work would be needed before one could be confident of the cut sizes obtained with cyclones operating at high temperatures.

Although the maximum diameters at which inertial separators provide size classification are only about 10 to 20 μm, the larger particles are collected so information on the total concentration including the largest particles present in the gas stream is obtained when they are used.

In order to deal with the extremely high temperatures associated with this study, some adjustments would have to be made from some combination of the following (or other) possibilities. (1) The samplers could be constructed with materials that can withstand such high temperatures, appropriate adjustments to the physical constants associated with the devices being operated at such temperatures would have to be calculated and verified, and procedures for handling the devices, processing the data, and calibrating the systems would have to be developed. (2) In situ probes could be cooled somewhat to allow the devices to operate at more tolerable temperatures in high temperature streams without long transport lines for the sample. (3) The sample gases could be extracted and cooled prior to introduction to the devices while minimizing and accounting for line loss during the process.

SECTION 3

RECOMMENDED METHODS

The ultimate objective of this project was to develop procedures that the ARB can use to measure particle size distributions at a variety of source types at which flue gas conditions would not permit the hardware and procedures of Method 501 to be employed. Inherent in this objective was the requirement that the procedures proposed be reasonably easy to use over a range of site locations and types.

3.1 Methods for High Temperature Sources

Approaches for sizing particles in high temperature effluent streams had to deal with the heat and its effects in one of a limited number of ways. One approach was to construct conventional inertial sizing equipment using materials that can withstand both the heat and its effects and the potentially corrosive nature of the flue gases. A second was to modify the measurement probe of some form of more conventional inertial or optical sizing equipment so that it could be cooled enough to perform its function as it extends into the hot gas stream, otherwise using standard materials and procedures. A third approach was to use optical devices that would physically located outside the stack and transmit the light through windows into the gas stream. Finally, there was the approach of extracting a sample and transporting it out of the stack, cooling it with or without dilution, and then using either conventional optical or inertial sizing devices. No special modifications or materials would be necessary using the latter approach in constructing the sizing devices themselves and commercially available equipment could be employed.

3.1.1 High Temperature Materials

All of the approaches mentioned above involve some exposure of the prospective sizing device to the high temperature environment in part or in whole, whether the entire instrument, a probe encasing the instrument, or an extraction nozzle and sample line. Efforts since the 1970's to develop particulate control and monitoring equipment for coal conversion systems led to studies on the limitations of commercial materials and the subsequent development of materials that resist deterioration under high temperature (>1200°F), high pressure, and sulfidizing or carburizing atmospheres (Hull, 1977). This current project does not address the high pressure environment, and the extent of sulfidizing or carburizing in the atmospheres to be encountered is not at all predictable. However, the high temperature findings are still relevant. Materials for high temperature sampling must be either metallic or ceramic, since polymeric materials cannot survive temperatures above 800°F.

Metallic materials are subject to compound formation, and can oxidize (or carburize or sulfidize). Alloys that resist oxygen at high temperatures do so by forming protective oxides. These oxides then act as barriers to further oxidation but do not effectively resist penetration by sulfur and carbon at high temperatures. Metals useful in high-temperature environments include the iron-base, nickel-base, and cobalt-base alloys. The iron-base alloys include both stainless steels and superalloys. While iron is the main constituent, chromium is an important alloying element present in significant quantity ($\approx$ 20 percent), and nickel generally is present. Chromium forms a tenacious, protective oxide barrier, and nickel adds significant high-temperature strength to the alloy. Nickel- and cobalt-base superalloys generally have nickel and cobalt as the predominant elements in the alloys, but they will often contain a substantial amount of chromium and iron (Hull, 1977).

Corrosive attack of a metal is highly dependent on the gaseous environment. Therefore metals should be selected for the specific environments to which they will be exposed. For instance, a high nickel alloy might be desirable in many high temperature environments for its strength; however, in a high sulfur environment, the nickel present may cause a catastrophic failure of the material because nickel sulfide will be a molten slag at temperatures above 1193°F. Modern superalloys such as Haynes 230 and Haynes 556 offer far superior resistance to oxidation, carburization and sulfidation than any of the stainless steel types, Hastelloy, and Inconel; hence they were recommended and selected to be used for the metallic portions of any chosen system(s) that would be in direct contact with the hot flue gases. Haynes 230 suffers oxidation losses at a temperature of 2000°F of 1/3 to 1/5 that of stainless steel, Hastelloy X and Inconel. Haynes 556 has shown similar performance on exposure to hot off gases from industrial waste incineration at temperatures of 1400°F to 1600°F.

Ceramic materials are made up of compounds such as oxides, carbides, and sulfides. Since these compounds must dissociate and recombine in order to form new compounds, they tend to resist corrosive attack more successfully than metals. Ceramics also are more resistant to high-temperature attack because they are more refractory than metals (their melting or dissociation temperatures are significantly higher). The erosive effect of particles impinging on ceramics is generally less severe than erosion on metals, especially at glancing angles. But they do have some undesirable properties, since they are weak in terms of tension and have poor thermal shock resistance. They are subject to attack by steam in the temperature range from 1000°F to 1700°F, where silica is leached out to form silicic acid. When the temperature exceeds 1700°F, ceramics are subject to hydrogen attack. High-density ceramics are preferable to low-density ceramics since their susceptibility to these attacks is less (Hull, 1977). One possible solution to the high temperature materials problem might be a literal merging of these two approaches. Ceramic coatings on metals were previously found to be too brittle to endure differential thermal expansion, but more durable ceramic coatings are now being offered in which chemical bonding with the metal lattice structure is achieved. Alternatively, diffusion alloying with metallic or intermetallic compounds result in surfaces that are quite resistant to abrasion, erosion, corrosion and oxidation. These surfaces are not glass brittle, will allow

some plastic deformation without cracking, and have no grain boundaries. Coatings of this type have been put on stainless steel, carbon steel, STELLITE, and Hastelloy X and interior surfaces and blind holes can readily be coated (Clark and Sievers, 1984).

Bill Powers of ENSR (personal communication, 1992) reported that PM_{10} cyclones constructed of 316 stainless steel did not survive use in flue gas at 1550°F. Since 316 SS has a design temperature limit of 1200°F this was not altogether unexpected. ENSR obtained drawings of the cyclones from SRI and had an Inconel version made. The latter was demonstrated to provide physically acceptable service at temperatures to 1600°F. Since there were more promising materials available than Inconel, the materials problem for making components capable of direct exposure to high temperature flue gases appeared to be tractable even in the absence of cooling provisions.

3.1.2 Cooling an In Situ Probe

In order to avoid the expense of using high temperature materials or to avoid having to deal with complexity of adjusting mechanisms, theoretical or empirical constants, or calibrations of existing equipment to adapt to the high temperature environment, some have opted to cool in situ probes. Even with optical systems operated outside a combustor, such as the particle-sizing interferometer (PSI) of Farmer et al. (1983), it was necessary to operate the instrumentation at much cooler temperatures than those of the flue gases. Therefore, the latter systems were fully enclosed and cooled with nitrogen purges. Beck and Hayhurst (1990) collected and quenched samples prior to off-line analyses by means of a water-cooled silica probe. When making measurements in high temperature or otherwise extreme environments, Bonin and Queiroz (1991) inserted their Particle-Counter-Sizer-Velocimeter Probe (PCSV-P) in a water-cooled jacket that could be extended into a sample gas stream with temperatures up to 2600°F. In the latter case, and with other in situ optical systems, the optical and electrical components of the sensor must be kept at normal ambient temperatures for them to survive. The probe developed by Masters et al. (1979) used a heat exchanger and recirculating Dowtherm fluid for cooling of both the probe shell and the impactor within it.

One of the biggest drawbacks with water or other liquid cooled probes is that the coolant and the pump used to provide it generally are too heavy and bulky to be easily transported to difficult to reach, and often hazardous, sites. Personnel of the ARB expressed strong reservations about the use of methods or systems that would require water cooling and, because other viable approaches were available, ideas for systems that required water cooling were dropped.

3.1.3 Extractive Systems

Note: Extraction as used here does not necessarily imply transport of the sample out of the duct. EPA Method 17 and cascade impactors operating in situ are both

examples of extractive systems with no subsequent sample transport. EPA Method 5 is an extractive system with transport.

There are several factors which may lead to the necessity of sample extraction, with or without some other form of conditioning, such as cooling. Montagna et al. (1977) tested two non-extractive light-scattering particle size analyzers on 1650°F flue gas, comparing their results with those of an Andersen cascade impactor, a Coulter counter, and absolute filters. Sampling with the impactor and filters was carried out with probes to extract the sample and cool it to temperatures that the measurement devices could withstand. However, the sample line was electrically heated to maintain the temperature of the gas sample above its water dew point. The particulate sampler for high-temperature, high-pressure processes developed and demonstrated by Masters et al. (1979) used an extractive approach, removing samples from the process stream for complete analysis of particle size, distribution, morphology, and chemical composition. A cascade impactor was used both to collect and to size particles. But the gas was cooled from the measured process conditions of 1350°F to less than 500°F, the maximum temperature for which variations in cut size with temperature had been verified for the impactor at that time, before the size separation took place. Self (1982) contended that extractive sampling had the advantage of allowing a wide range of available instruments to be brought into use. However, he cautioned that care must be taken to avoid biasing the distribution by non-isokinetic sampling and by size-dependent loss of particles in flow lines and dilution/quenching devices. Extractive sampling from high temperature flows posed additional problems since quenching of the flow may result in condensation of water and mineral vapors. Wang and Libkind (1982) extracted the sample for the particulate mass analyzer they developed and demonstrated on a 1520°F gas stream. They kept the sampling probe and cyclone train heated electrically to 1520°F to keep the sampled gas from cooling. The reason they extracted the gas was that the tapered-element, oscillating microbalances (TEOM) used in the analyzer had to be kept below 440°F.

Even among the optical techniques, single particle analyzers may require a more restricted sample volume than the diameter of the stack. Some optical systems are rather bulky, making cooling for in situ operation impractical and require working distances from windows that are too short to permit operation across a duct or stack. In the two-color laser Doppler technique reported by Hemsley et al. (1985), the sensing volume could not be located within the duct, consequently the gas sample on which the particle size measurements were performed was removed isokinetically from the stream. Many, if not most, of the devices used to measure ultrafine particles have some limitations on the particle density and temperature range they can handle without significant errors (Felix et al., 1981; Pyle and Smith, 1984). Therefore, some form of a sample extraction, conditioning, and dilution system is required for them under most field conditions and certainly would be for the conditions of interest to the ARB in this work. Heiskanen and Kauppinen (1989) reported on one such system to measure size distributions of oil combustion aerosols. Beck and Hayhurst (1990) extracted samples of char leaving premixed flames for chemical analyses by a filter assembly. There was concern about

reaction between the char and the air once it had cooled before analysis. Chemical washing was necessary to remove the char from the filter, requiring that proper solvents (ones that would not adversely affect the filtrate) be used. The particles had to be suspended in yet another liquid for particle size analysis. Mild ultrasonic treatment was necessary to ensure proper dispersion of the material and to break up any agglomerates that may have formed. The particle size analyzer used was a Malvern Instruments ST 2200, which employed a method of laser forward-scattering. Samples with broad size distributions gave erroneous results, in that the proportion of large particles was underestimated. Therefore, all samples were sieved quantitatively to remove the majority of smaller particles for separate analyses. Fournier et al. (1990) collected particulate samples from an afterburner exit flue of a kiln using a variation of a Method 17 sampling train. These particles were collected in an oversized alundum thimble and later divided using a Bahco Micro Particle Classifier. Hsu et al. (1991) extracted and analyzed fume particles using a scanning electron microscope (SEM) to compare the results obtained with their studied optical technique.

3.1.4 Selected Methodology for the High Temperature Application

For measurements at the high temperature sources, the optical techniques were attractive because many of them are non-intrusive and near real-time in analysis. However, they had major drawbacks in their lack of response in the small particle size range, which was one of great interest, and their potential lack of accuracy due to refractive index uncertainties for the larger particles. The latter can lead to very large errors in mass concentrations derived from optical data. Many of the potential optical systems had lower limits of detection of 2 to 5 μm, which were not small enough to detect many of the respirable particles. Others with lower detection limits still biased toward larger particles in a heterogeneous distribution, often with the small particle signals being lost in the background noise of the larger particle signals. Varying degrees of dependence on index of refraction (real and/or complex) and on the assumption of spherical shapes may also tend to make some optical results questionable. Some of the single particle analyzers have shown potential or capability of measuring in the smaller range, but many of these require small sampling volumes in order to accomplish this. Such small volumes require either a cooled probe or an extracted sample, both of which somewhat negate the non-intrusive argument. And small sampling volumes limit the ability to measure large particles because they are relatively much less numerous, resulting in large statistical uncertainties in their count rates. Moreover, most of the optical techniques are moderately to quite expensive, intricate in their mechanisms, and limited to restrictive physical requirements (clean, non-distorting optical ports). And there was some question as to whether their application may be flexible enough to cover the wide range of high temperature source types and conditions that were of interest to the ARB.

If optical methods were eliminated, the most promising techniques were the inertial classifiers. Inertial methods have been used in a wide variety of applications; so their versatility is assumed to be adequate. Although they do not provide classification at large

sizes, they do include all sizes present in a representative fashion. The high temperature environment poses a number of new conditions, however. Construction of existing designs with high temperature materials would seem to be one logical solution. However, finding reliable, non-reactive substrates for cascade impactors that can withstand such temperatures was also quite problematic. Water cooling the impactor and sample lines would alleviate these problems but the added weight and logistical difficulties make this option unattractive to the ARB just as was the case for optical probes. Extraction, transporting the sample out of the duct, and cooling would eliminate this objection, but would introduce the problem of deposition of particles in the extraction lines, especially for the larger sizes. However, the latter problem could be dealt with in a reasonably satisfactory manner as will be discussed below.

Thus in the final analysis, the use of modified versions of the current ARB particle sizing method was recommended. This approach had several advantages in that much of the presently used hardware could be employed and the results obtained would be on the same basis as that which was used to build the existing database. Three modified methods were suggested, one for each of three application conditions.

3.1.4.1 Moderately high temperature applications

For flue gas streams with temperatures below 800°F the current ARB particle sizing method of inertial separation using cascade impactors can be employed. The only change from the current method is that quartz fiber substrates and filters be used for the collection media. Several years of field experience at SRI have shown that the University of Washington (Pilat) cascade impactors can be used to 800°F with teflon tape as a thread sealant and lubricant and Viton O-rings for inter-stage seals. The teflon tape will liquify but will still provide the needed external seals and the Viton O-rings will provide good interstage sealing. However, the O-rings, when used at these temperatures, will harden too much for repeated use and can be used for only one sampling run after which they must be discarded and replaced. The quartz fiber collection materials provide suitable non-reactive impaction surfaces and filter media that are usable at temperatures to well above 800°F. No hardware development was required for applications of this type. Further information regarding the design and calibration of the UW (Pilat) impactors has been given by McCain et al., 1986a,b.

3.1.4.2 Very high temperature ducted sources

For sampling of ducted emissions at temperatures above 800°F the system shown in Figure 3 was selected and, with the approval of the ARB, a working, field-usable prototype was constructed. The prototype was designed for operation only with the impactor mounted externally (location 2 in Figure 3). Because it must operate at flue gas conditions, a high temperature version of the right angle precollector used in the current ARB particle sizing method was constructed of Haynes 230 alloy to provide the first stage size separation. The remainder of the system consists of a cylindrical probe sheath also made of Haynes

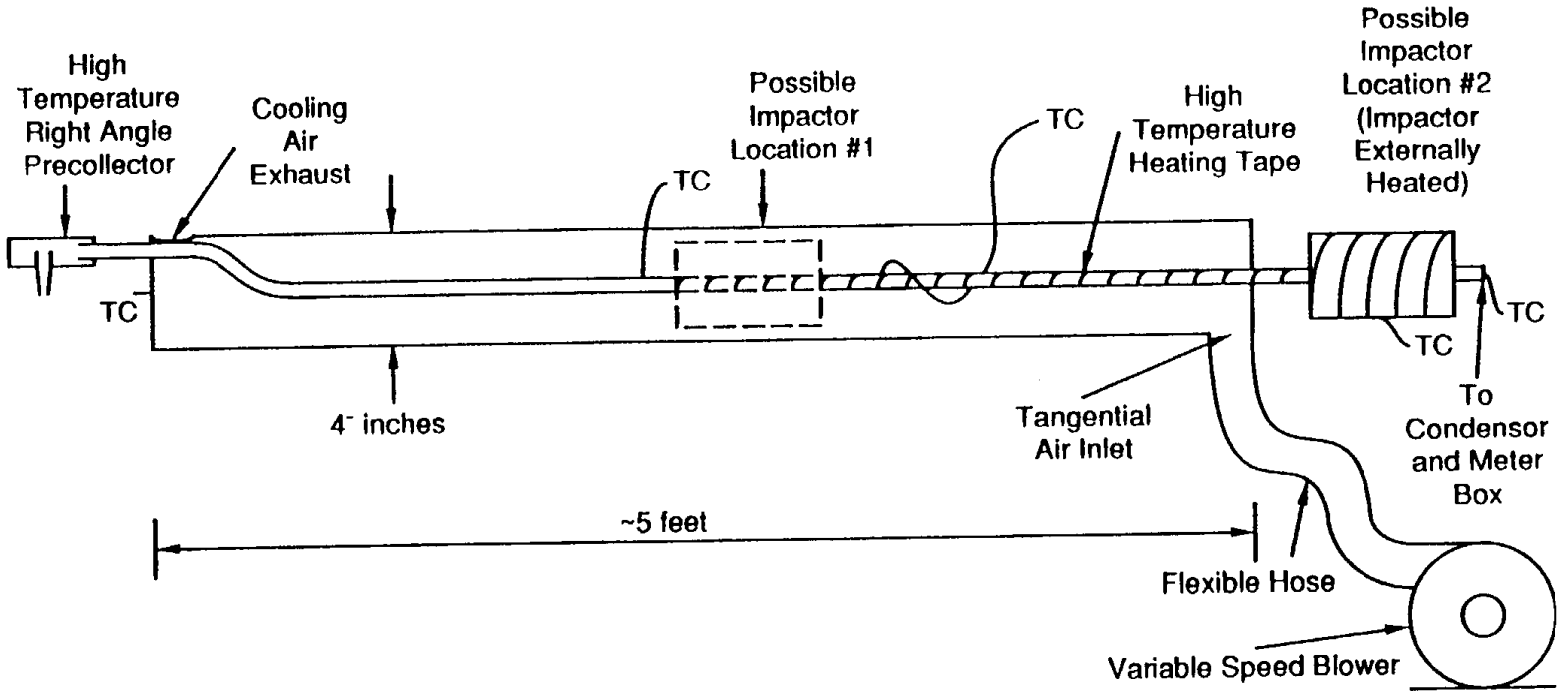


Figure 3. Layout of the ARB sampling system for ducted high temperature particle size distribution measurements. Details of the preimpactor may be found in Appendix B.

230 alloy that contains a sample transport line made of Haynes 556 tubing about four feet in length. The internal cavity of the probe is cooled with ambient air provided by a variable speed blower. A suitable blower weighing less than 10 pounds was selected for this application. Air cooling cannot reduce the temperature of the probe and sampled gas nearly as much as water. However, unlike optical systems, the impactor does not require near ambient conditions to survive or function. Reducing the operating temperature of the impactor to below 800°F is sufficient to permit relatively trouble free operation. Thus the weight, complexity, and logistical problems associated with water cooling could be avoided. The cooling air is simply allowed to exhaust into the flue gas stream in the downstream direction from the side of the probe opposite the sampling nozzle. Because the impactor is not exposed to the very high temperatures of the duct, special construction materials are not needed for it and standard, off-the-shelf, hardware can be used.

Projections of the external sheath and internal cooling air temperatures for such an air cooled probe are shown in Figure 4 and 5. The precollector removes the larger particles from the sample, those at greatest risk of line loss in extraction techniques, in a predictable and consistent fashion and operates as the first impaction stage. In the prototype the impactor itself is located a few feet back from the precollector, outside the stack and the probe sheath. A heating jacket or heating tape is used to heat the impactor to the desired operating temperature. A silicone rubber insulated heating jacket was provided for this purpose with the prototype system. The latter is limited to temperatures below 450 °F. If higher operating temperatures are deemed desirable, a Samox type heating tape can be used to permit operation at temperatures to 800 °F, the nominal limit for the impactor seals. Operation at a temperature of 350 °F is recommended for routine sampling.

If applications are encountered that require a long probe, a variant of the prototype could be made with a sheath that has a larger inside diameter than the one actually fabricated. This would permit the impactor to be mounted inside the sheath, still using a few feet of tubing to make the connection to the precollector. In this case the temperature of the impactor, located within the probe, will be intermediate between the outer sheath wall temperature and the temperature of the cooling air. Locating the impactor within the sheath in this case is desirable because this permits the use of a relatively short transport/cooling tube, thus minimizing transport losses between the precollector and the impactor.

The precollector used for the prototype probe was designed to provide an effective size cut, or D_{50} that is as large as possible and as little influenced by the nozzle tip diameter as possible while still permitting operation through a four-inch diameter sampling port. The gas velocity must be reduced as much as possible between the nozzle tip and the first turn the gas and particles make to maximize the D_{50}. The total included angle at which the flow will diverge is about 7 degrees (total included angle or a 3 degree half-angle), so the only way to affect any substantial drop in velocity is to make the deceleration distance as large as possible. This requires a geometry with relatively large internal dimensions,

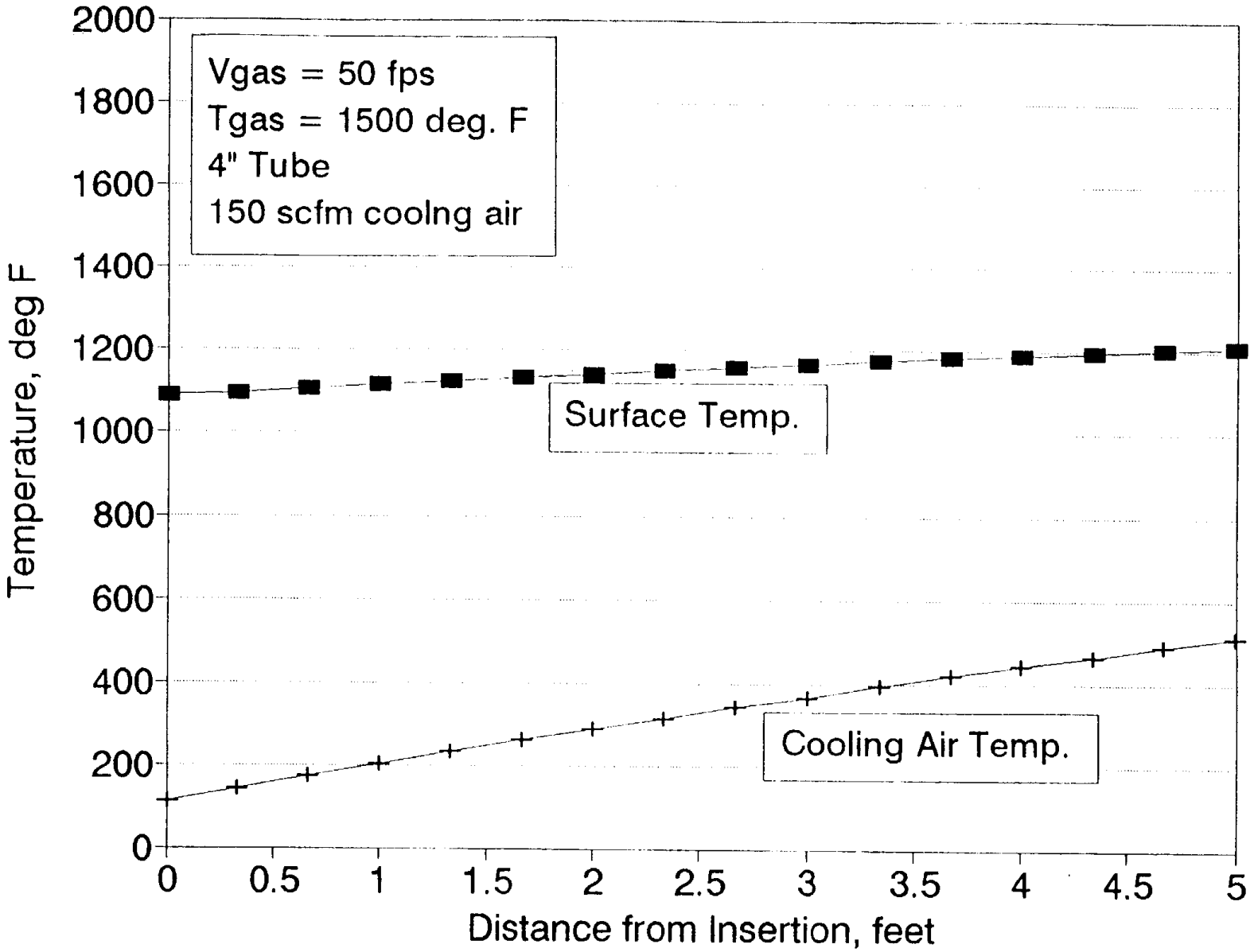


Figure 4. Estimated probe surface and internal air temperatures for a four inch diameter high temperature particle sizing probe operating in a 50 foot per second, 1500°F gas stream.

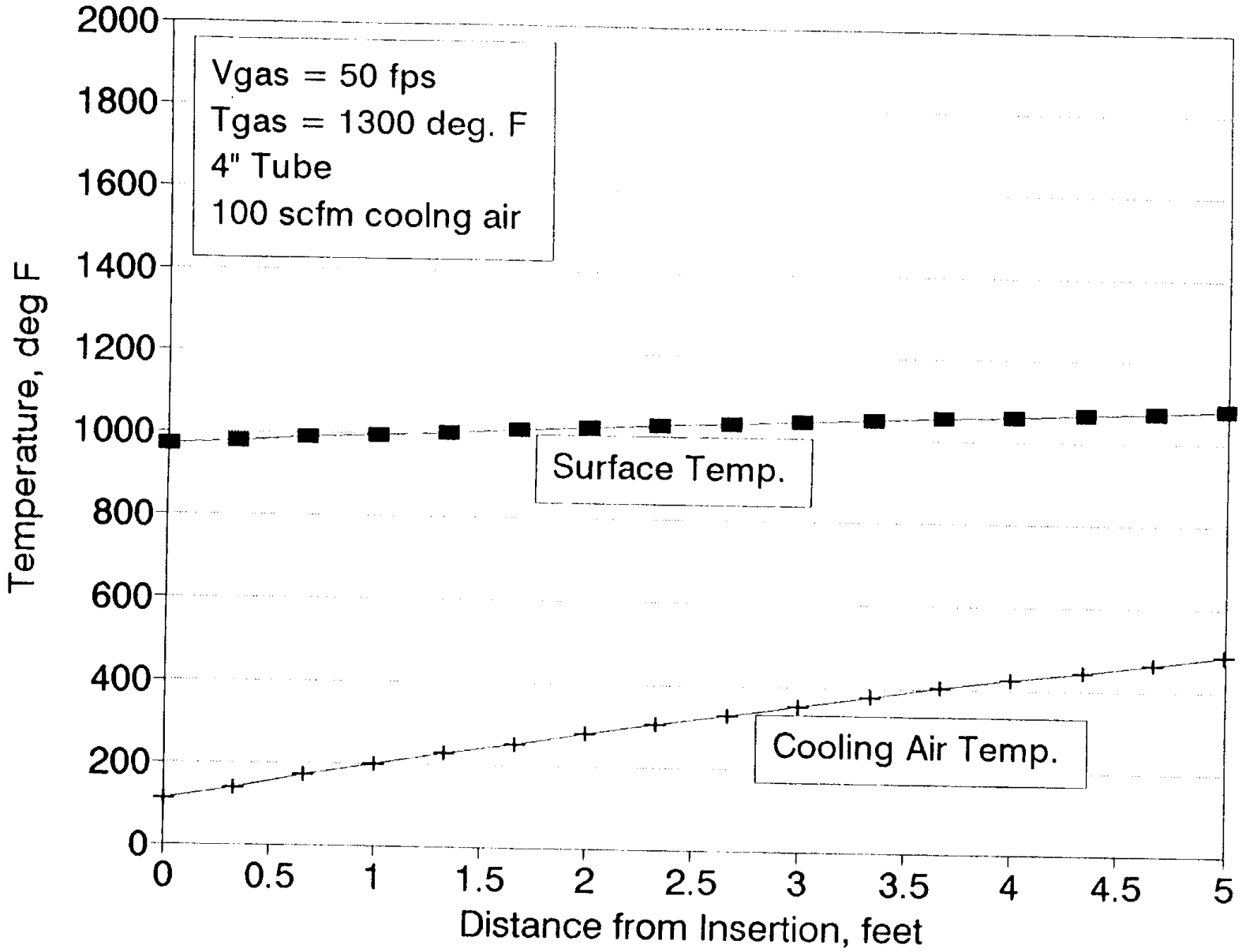


Figure 5. Estimated probe surface and internal air temperatures for a four inch diameter high temperature particle sizing probe operating in a 50 foot per second, 1300°F gas stream.

minimal expansion angles, and as long a deceleration length as possible downstream of the nozzle tip. Figure 6 shows the calculated transmission efficiency of the precollector alone and the precollector followed by a five foot long, 0.5 inch ID sample line. The actual prototype probe was constructed with a four foot sample line, so losses in it would be slightly lower than those shown in Figure 6. Calibrations for the SRI/EPA Right Angle Precollector (RAPC) are provided in Appendix C together with predicted values based on a semi-empirical theory developed at SRI for such devices. The sharpness of cut obtained with the SRI/EPA RAPC is comparable to that of conventional impactor stages. The high temperature precollector designed for the ARB was based on the SRI/EPA design, but used longer nozzles to allow greater velocity dissipation and, consequently, permit larger precollector D_{50}s to be obtained.

The precollector performance prediction was based on current calibration data that was obtained largely at ambient conditions. Therefore some experimental verification will be needed for use at very high temperatures. The model used to predict the precollector performance and transport efficiencies in the tube from the precollector to the impactor shown in the figure was one recently developed for the US EPA in conjunction with a program for the development of continuous particulate mass emission monitors. It includes the effects of losses in bends, losses from gravitational settling, losses from Brownian diffusion, and losses from turbulent deposition. As the figure shows, transport losses from the precollector to the impactor, can be expected to be virtually negligible at the sampling rates that are anticipated under normal usage conditions. However, settling losses can become important at low sampling rates if the probe is horizontal.

Heaters must be provided along the extraction line and around the impactor to keep the temperature above the moisture dew point. Two independently controlled high temperature heating tapes are used inside the sheath of the prototype probe. The first, at the exit end of the probe, is tightly wound and insulated so as to prevent excessive cooling near the cooling air inlet. The second is a more loosely wound heater that partially covers the remainder of the sample tube up to a point near the precollector end of the sheath and serves primarily to provide a means of preheating the sample tube. However, it also serves to keep excessive cooling from taking place in instances in which the insertion distance is not great enough for the cavity to be heated by the flue passing over the sheath. The heating tapes used inside the sheath are rated for temperatures up to 1600 °F and the alloys used are rated to temperatures of at least 2500 °F. The cooling air will provide sufficient cooling for the probe to be used to temperatures of at least 2000 °F. Operation at higher temperatures may well be possible but caution should be used at stack temperatures in excess of 2000 °F.

3.1.4.3 Non-ducted high temperature applications

For sampling open flares and similar sources the principal difficulty will be that of mechanically positioning the sampling system at a suitable location in the effluent stream from the flare. "Cherry picker" type of lifting devices have been used to provide access for Method 5 type sampling of flares in the past and should be satisfactory here as well.

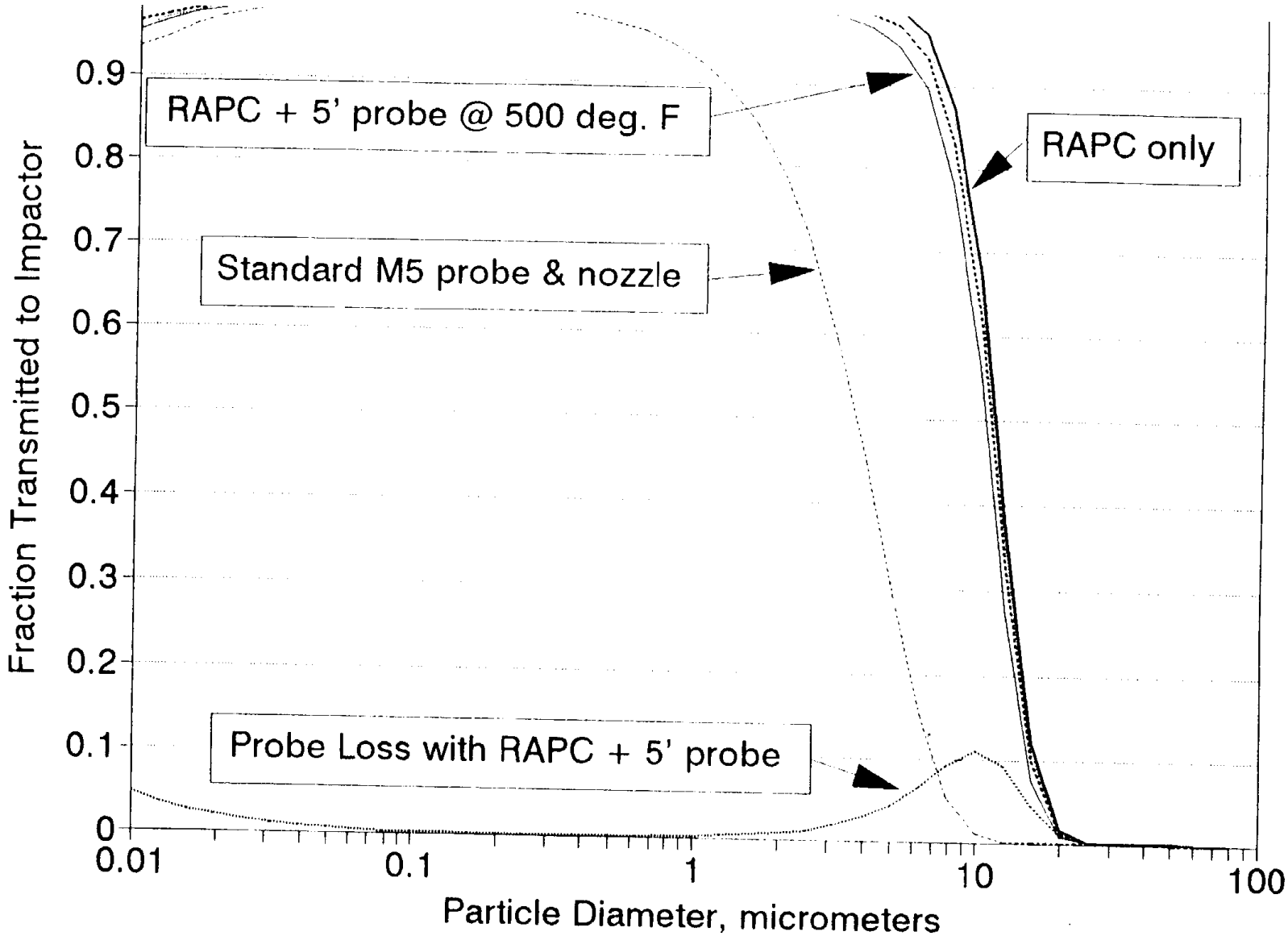


Figure 6. Estimated particle transmission versus particle diameter for a high temperature probe consisting of right angle precollector (RAPC) and a five foot, 0.5 inch inside diameter, transport tube. The transmission curves were calculated for a flow rate of about 0.25 scfm with the RAPC operating at 1500°F and gas temperatures through the tube of 1500°F, 700°F, and 500°F. The probe loss curve shown is for a temperature of 500°F.

Because the high temperature zone in/above a flare will have a relatively small diameter a simpler approach than the sheathed probe can be used. If the absence of particles in the emissions larger than about 10 μm can be demonstrated, a simple, short, unheated length of superalloy tubing followed by an additional few feet of heat traced tubing to a heated impactor will suffice as a probe. The initial length of exposed tubing provides for some cooling of the sampled gas while the additional heat traced portion moves the impactor (and operator) back away from the high temperature zone. Provided large particles are not present (say bigger than 10 μm), losses in the probe would be acceptably low as was shown in Figure 6. And if only fine particles, say 3 μm and smaller are present, isokinetic sampling would not be required. If larger particles are found, and especially if particles larger than about 10 μm are present, isokinetic sampling would be required and the high temperature right angle precollector would be needed but the sheathed cooling system could probably be dispensed with.

3.1.4.4 High temperature collection media

Inconel or superalloy metallic shim might be used as collection substrates in the precollector or, alternatively, Fiberfrax or Kaowool papers might be used. The properties of one of these, Kaowool, are provided in Table 3.1. All of these materials have been used as impactor substrates at temperatures over 1200°F by SRI in work done for the US EPA and DOE. Fiberfrax and Kaowool papers are materials much like glass filter paper, but are made from silica, alumina, and/or ceramic fibers that will survive high temperature environments. SRI and other organizations have successfully used Fiberfrax for impactor substrates and backup filters in samplers operated at temperatures up to at least 1200°F. The substrates and backup filter for the impactor can be conventional quartz fiber materials (eg. Pallflex QAST 2500 Tissuequartz). These have been demonstrated to perform satisfactorily in our own field work to temperatures of at least 800°F and are rated by the manufacturer to be serviceable to 1500°F.

Detailed procedures for using the proposed method are provided in Appendix B, "M501b - Particle Size Measurement for High Temperature Sources."

3.1.5 Operational Testing of the High Temperature System

Two sets of operational trials were conducted using the prototype high temperature system. The first was a shakedown test carried out at Southern Research Institute's pilot scale coal combustion facility and the second was carried out as a demonstration test for ARB personnel carried out at a landfill flare located in Southern California. Each of these will be discussed in turn.

Table 3.1 Physical Properties and Chemical Analysis of Kaowool				
Physical Properties	Rollboard	Paper		Ultrafelt
		2300	3000	
Color	gray	white	white	white
Density, lbs./cu.ft. (Kg/m^3)	11-14 (176-224)	11-14 (176-224)	10-12 (160-192)	8-10 (128-160)
Maximum Temperature rating, °F (°C)	2300 (1260)	2300 (1260)	3000 (1649)	2300 (1260)
Thickness, inches (mm)	1/10 (2.54)	1/16, 1/8, 1/4* (1.58,3.17,6.34)	1/32,1/16,1/8* (.78,1.58,3.17)	1/10,1/4* (2.54,6.34)
Chemical Analysis: (Typical Weight, 0/0 After Firing)				
Alumina, Al_20_3	47	47	94	47
Silica, SiO_2	53	53	6	53
Other Inorganics	Trace	Trace	Trace	Trace
Loss on Ignition (LOI)	5-7	5-7	8-10	-

3.1.5.1 SRI coal combustor tests.

Shakedown trials of the high temperature system were performed at the SRI coal combustion facility on January 25 and 26, 1994. On January 25 the system was used to sample coal combustion flue gas at a temperature of 1110°F. No major problems were encountered during that test but system temperature measurements led to the conclusion that a greater length of the probe inside the sheath should be heated and insulated than was done for this test. The heater and insulation were reconfigured and a second trial was performed, this time at a location on the combustor where the gas temperature was about 1500°F. No problems were encountered during this test and the system was deemed to be performing as intended. Detailed operational data and results are provided in Appendix D.

Table 3.2 provides records of temperatures through the system during the preheat period before insertion into the duct, during the stabilization period in the duct prior to sampling, and during sampling, for each of the two sampling runs. The columns identified as "Probe" in the table refer to the part of the sample tube inside the sheath. Because of the small dimensions of the combustor ducting, a portion of the sample tube that would normally project into the duct had to be retracted so that the unit could be tested with an appreciable part of the sheath in the duct. This resulted in a corresponding length of the sample tube at the exit end that would normally be inside the sheath extending outside it by about 10 inches. A separate auxiliary heater was used to maintain the tube temperature along this external length of tube. The latter is what is what is referred to as "Tube" in the table.

As expected, about one hour of preheat was needed before the impactor and the part of the probe that would be external to the duct had reached temperatures that were high enough and sufficiently stable for the system to be ready for inserting the probe into the duct. An additional period of about 45 minutes was needed after insertion for the temperatures to stabilize well enough to begin sampling. A much longer wait after insertion was used in the second test, but that was because of delays to allow other combustor operations to take place before sampling was started and was not dictated by the sampling system.

The combination of very low gas velocities (approximately 12 fps) and high particulate loadings at the two sampling points resulted in sampling flow rates being used that were substantially lower than those anticipated during the design of the system. (Nozzles sized for isokinetic sampling at higher sampling rates for low velocity streams were not made for the prototype unit because of cost considerations. The gas velocities commonly encountered in the field are in the range of 30 to 100fps.) For that reason, particle deposition in the probe was greater than would normally be the case when the system is operated on relatively clean stacks with more normal (higher) gas velocities. The high probe deposition resulted from two factors. First, the low sampling rate resulted in precollector cut diameters that were larger than would have been obtained at higher

TABLE 3.2

Probe Heater and Blower Power Settings and System Temperatures during SRI Shakedown Tests of the Prototype HT Particle Sizing Probe

HT Probe Run 1 1/25/94

					Gas Floor through Sampling System = = > >						
	Variacs, % of Maximum				Temperatures						
Time	Imp.	Tube	Probe	Blower	Stack	Probe 1	Probe 2	Probe 3	Probe 4	Tube	Impactor
1420	40	40	50	Begin Preheat		89		262	501	178	176
1425	40	40	40		1109	93		290	535	251	221
1445	40	40	36			103		318	582	339	292
1453	40	40	36			106		311	552	356	
1510	36	40	36								
In: 1526	30	35	36			120		260	513	392	390
1532	30	35	45		-1110	242		165	366	373	378
1538	30	35	0	(unplugged)		406		115	198	365	382
1550	30	35	55			412	144	155	374	369	385
On: 1600	30	35	55		1116	447	168	228	635	380	375
1602	30	35	55			495	*258	258	653	384	377
1604	30	35	55			512		256	602	384	377
Off: 1605											

*Led to decision to heat and insulate more of sample tube in sheath blower not required for this run - cooling air inlet had to be blocked to keep excessive cooling from taking place - duct was at negative pressure.

Ht Probe Run 1 1/25/94											
1400	30	40	40	Begin Preheat							
1630	35	35	35			104	128	313	452	350	367
In: 1739	35	35	35	0	1480	153	135	314	432	330	355
1741	35	35	35	0	1480	283	188	305	415	330	352
1744	30	40	46	25		482	290	300	400	331	348
1747	30	40	46	55		521					
1754	30	40	46	55	1507	543	300	264	354	328	344
1756	38	40	46	55							
1800	38	40	46	55	1486	567	343	260	338	326	356
1810	36	44	44	55							
1815	44	44	36	55	1466	556	318	256	320	327	379
On: 1830	36	44	44	55	1452	559	347	269	326	335	369
1835	36	44	44	55	1453	589	380	334	375	330	372
Off: 1840	36	44	44	55	1455	585	380	326	388	331	374

sampling rates. This meant that a greater proportion of the larger particles, which have greater settling velocities, were passed to the transport tube. And second, the lower flow rates through the transport tube allowed more time for settling to occur, increasing the minimum size for which probe deposition would be significant.

As previously mentioned, SRI developed a computer model under contract to the US EPA for predicting particulate deposition in probes and precollectors like those developed under this contract for the Air Resources Board. That was the model used during the design work on this contract to assess the potential affects of deposition in the sample lines on measurement results. This model was applied for the sampling circumstances during these two shakedown runs. The operating conditions of the sampling system and the size distribution measured with the system were used as inputs to the model, which then provided predictions of the percentages of the particulate matter that would be expected to be found in the precollector and in the probe. The predictions for the precollector were 62% and 44% respectively for the two tests and the measured values were 60% and 46% respectively. The corresponding predictions for deposition in the probe were 35% and 32% while the respective measured values were 33% and 30%. In each case, the agreement between the measured and predicted values was quite good. The model also showed that the particles that deposited in the probe were of sizes that would have been collected by the first stage of the impactor in the absence of the cooling/transport tube. Thus the material deposited in the probe should have been and was considered as being part of the impactor's first stage catch for the purpose of analyzing the data. This can generally be expected to be the case.

At the higher sampling rates that are expected under normal conditions of use for the system, much smaller fractions of the total particulate matter sampled would be expected to be deposited in the probe. Such material, unless there are compelling reasons to the contrary, should be recovered and considered to be part of the catch of the first stage of the impactor. The proposed procedures drafted by SRI for the ARB call for the probe to be washed in a similar manner to a Method 5 probe and that the material recovered from the probe normally be considered as being part of the first stage catch of the impactor.

3.2 Methods for High Moisture Sources

While the high moisture scenario is quite different from the high temperature one, many of the basic principles are the same. One of the biggest differences is that, in the stack flow, there will be attached water around at least some of the particles. It is the assumed here that the particulate state of interest is the dry condition which would prevail downwind of a source after evaporation of the water, rather than the wet droplet condition in the duct. However, recommendations are also given for droplet size analysis should there also be a need for such measurements.

Water drops in stack gas streams typically originate from three sources:

Condensation on particles within the stream if the stream is at or near saturation conditions. These are generally smaller than 20 μm in diameter.

Small droplets which penetrate the mist eliminator of a scrubber. Again, these are generally smaller than about 20 μm in diameter.

Large droplets formed by spray nozzles or mechanically entrained from mist eliminator surfaces in scrubbers and from wet structural and duct surfaces. These droplets are typically several tens to hundreds of micrometers in diameter.

3.2.1 Large Droplets

Stopping distances for droplets of various sizes at two gas velocities, 10 and 20 m/s, are illustrated in Figure 7. The stopping distance of a particle is the distance it will travel in still air when it has a given initial velocity.

The significance of the stopping distance here is that it is impossible to effect a change of direction of 90° in a distance less than the stopping distance. The dimensions of practical sampling system components are on the order of a few centimeters at most, making it impossible to use an extractive technique to deal with droplets having diameters larger than about 15 to 20 μm. In practice, this means that it will not be possible to measure the size distribution of the large (>20 μm) droplets on either a wet or dry residue basis using an extractive technique. On the other hand, extractive systems show promise for diameters smaller than about 20 μm.

Because large droplets can be measured only in situ using a non-extractive method, it will not be possible to rigorously apportion the dissolved and suspended solids contained in them to arrive at a dry particle sizes which might result downwind. However, by collecting a representative sample of the large liquid droplets as a whole, the total dry residue associated with droplets larger than some known size can be determined. If measurements are made simultaneously of the liquid droplet sizes, estimates of the size distribution of the dry residue particles can then be calculated by assuming that the dissolved and suspended solids were homogeneously distributed in the original drops and calculating the dried sizes of the particles from the volume reduction that would result from the evaporation of the water. The technique for collecting the large drops is described later as part of the discussion on how to deal with droplets smaller than 20 μm.

If very large (250 μm or larger) droplets are present, the Electric Power Research Institute has found that a video droplet analyzer (VDA) designed by Southern Research Institute is the only field usable system that will give reliable droplet size and concentration information (Jones et al., 1990). The useful sizing range of the VDA in its most common

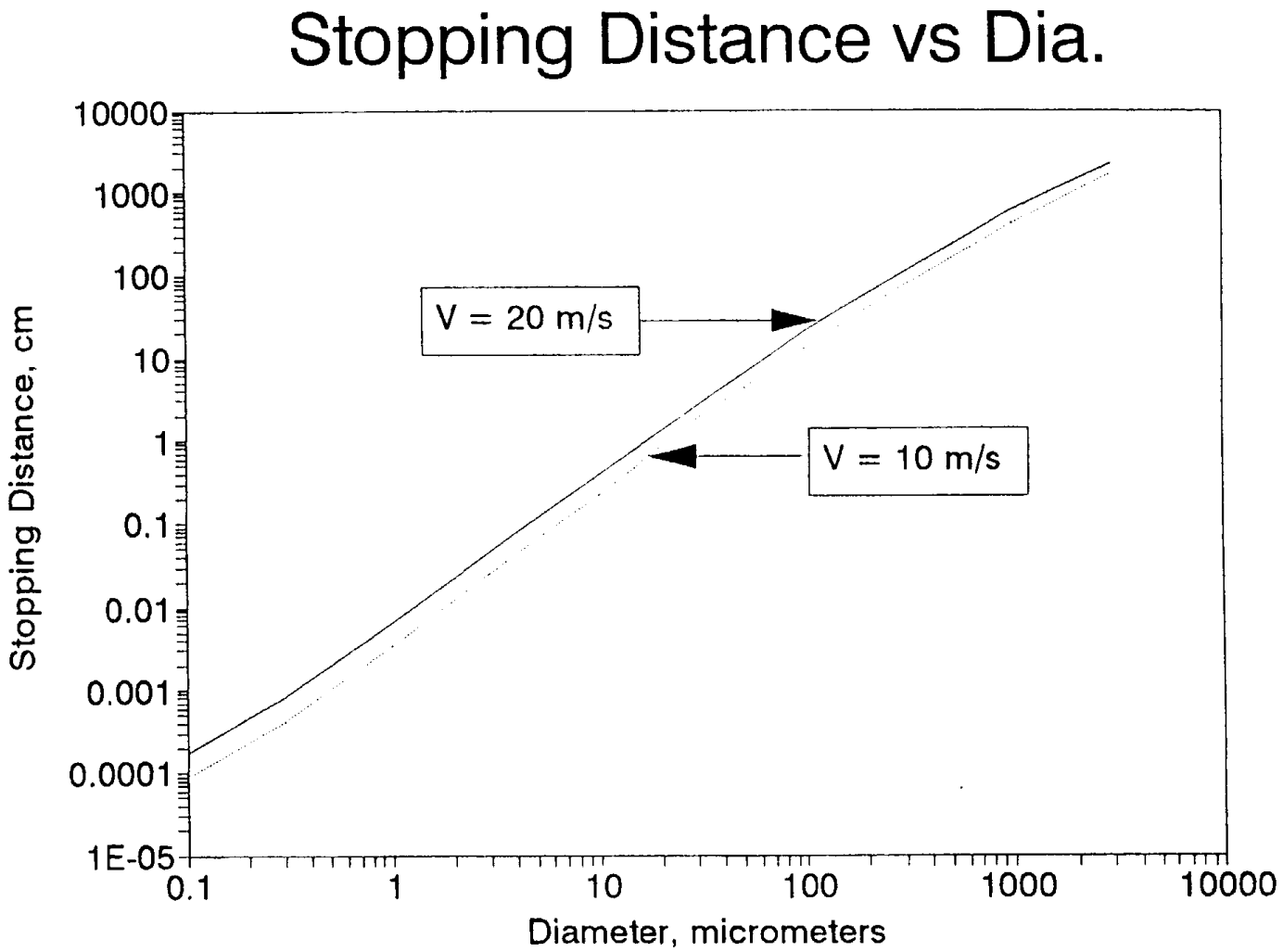


Figure 7. Particle stopping distance versus particle diameter for initial particle velocities of 10 and 10 meters per second.

operating mode is 20 to 3500 μm, but it can be configured for other ranges having the same relative limits. This device has a high capital cost, is somewhat unwieldy, and is fairly expensive to operate, requiring a trained three person test crew. The Koch corporation of Witchita, KS has also been reported as having an optical system for measuring droplets in stacks, but details about that system were not available to the authors of this report at the time it was being written.

If it is known that no droplets larger than about 250 μm are present, the AIMS hot wire droplet sizing device made by KLD Associates of Huntington Station, Long Island, NY, can be used to measure the drop size distribution (Magnus and Mahler, 1979). The latter device has much lower capital and operating costs than the VDA and has been shown to yield good results (Jones et al., 1990). This device operates by measuring the energy required to evaporate droplets which collect by impingement on a short fine wire (5 μm dia. by 1 mm long) rather like that of a hot wire anemometer. The sensing wire is mounted on the end of a light weight probe and is inserted into the droplet laden gas stream. Its sizing range is 1 to 250 μm. It can be operated by a single individual. The delicate nature of its sensing element makes sensor fouling and breakage a problem, but the elements are fairly inexpensive and easy to replace.

3.2.2 Condensation Aerosols and Small Drops

For sizes smaller than about 20 μm inertial sizing is again recommended. However, if one is interested in the size distribution of the residual solids after evaporation of the water, then something must be done with the water. As previously mentioned, droplets much larger than about 20 μm are too large to be effectively sized in even the first stages of the inertial separators, largely because the momentum of the droplets is too great to be stopped in the confines of a device that can be inserted through a port of practical dimensions as was shown in Figure 7. Therefore, before any effort is made to size the sample inertially, one must eliminate those droplets larger than about 20 μm. The most generally accepted way of accomplishing this task is through the use of an inertial precollector.

Cooper (1976) discussed solutions to the problem of removing and drying water droplets that are peculiar to sampling from scrubbers used for particulate and/or gaseous emissions control. He suggested that one might use special purpose preimpactors front ends of cascade impactor sampling systems for measuring the collection efficiencies of scrubbers by particle size. The preimpactors would be designed with relatively large particle size cut-offs to remove the larger water drops before the sample entered an impactor where the remaining particles/droplets could be sized. The right angle precollector now commonly used as a front end for inertial particle sizing with impactors grew out of Cooper's concept for such devices. An extension of this same technique is suggested as a means of measuring the size distributions of wet stack emissions as they would be found downwind after evaporation of the water.

Usually scrubbers are fitted with entrainment separators and mist eliminators that remove droplets that are larger than 10 to 20 μm in diameter. Thus large scrubber generated droplets are not likely to be present at a site with a well designed and maintained scrubbing system. If liquid is re-entrained from the scrubber, duct walls, or mist eliminators, it will be in the form of droplets that will be greater than 100 μm in size with typical sizes of several hundred micrometers. These would be removed by the preimpactor when present in a stream to be sampled. Normally, scrubbers would be expected to be quite efficient for removing incoming particles down to 5 μm diameter or so, thus capture by the precollector of the particles that penetrated the scrubber would not be a problem. The other likely source of droplets in the gas stream at wet sources is condensation. Although condensation droplets can be as large as 20 μm, they are typically smaller than 10 μm in diameter and would pass the precollector. Thus a high concentration "fog" of condensed water probably represents the most difficult case for a precollector/evaporation-by-heating front end for a cascade impactor system and, as will be discussed, sufficient heat and residence time can be achieved to make the technique usable.

The recommended method, then, for sizing particulate emissions from wet sources is to use a precollector to remove larger than about 15 to 20 μm. The residue, after drying, of the material collected in the precollector provides the value of the solids concentration associated with particles and droplets larger than the precollector D_{50}. Thus this provides the information needed to estimate the concentration and size distribution of the dry residue from large droplets when combined with the data the in-situ droplet sizing devices discussed in the previously. The water contained in the residual smaller droplets not removed by the precollector is then evaporated by passing the sample through a heating zone before the dried particles enter the impactor as illustrated in Figure 8. This requires both transferring enough heat to the gas to evaporate the residual droplets and giving the drops sufficient time to evaporate before reaching the impactor.

From equations given by Cooper (1976), one can calculate the required heating rate and time needed to evaporate aqueous droplets. Typical sampling rates for cascade impactors are about 0.5 to 1 acfm. Field experience by SRI in measuring the "dry particle size distributions" of emissions from scrubbers has shown that heating the sample gas stream to a temperature about 20°C above the saturation temperature in the duct is adequate for drying the droplets passed by a precollector of the type described here. The horizontal line in Figure 9a represents the heating rate (about 27 cal/sec) required to raise a 1 cfm flow of stack gas 36°F (20°C), assuming a mass equal to 10% of the mass of the gas is water droplets. The family of curves estimates the heating rate of different lengths of heating tubes with the tube wall heated to the indicated temperatures. As is shown, for a flow rate (1 cfm) and a 36°F increase in gas temperature, it would take an 18 in. heating tube with a wall temperature of about 800°F to supply the heat required to dry the droplets. If the flow rate is decreased to 0.25 cfm, with the other variables the same, the required heat input drops to about 7 cal/sec and shorter tubes and/or lower wall temperatures will suffice (see Figure 9b). This graphical analysis is based on the heat balance required to evaporate the a liquid phase mass equal to 10% of the gas mass flow. Smaller concentrations of liquid water would require less heat input (temperature rise).

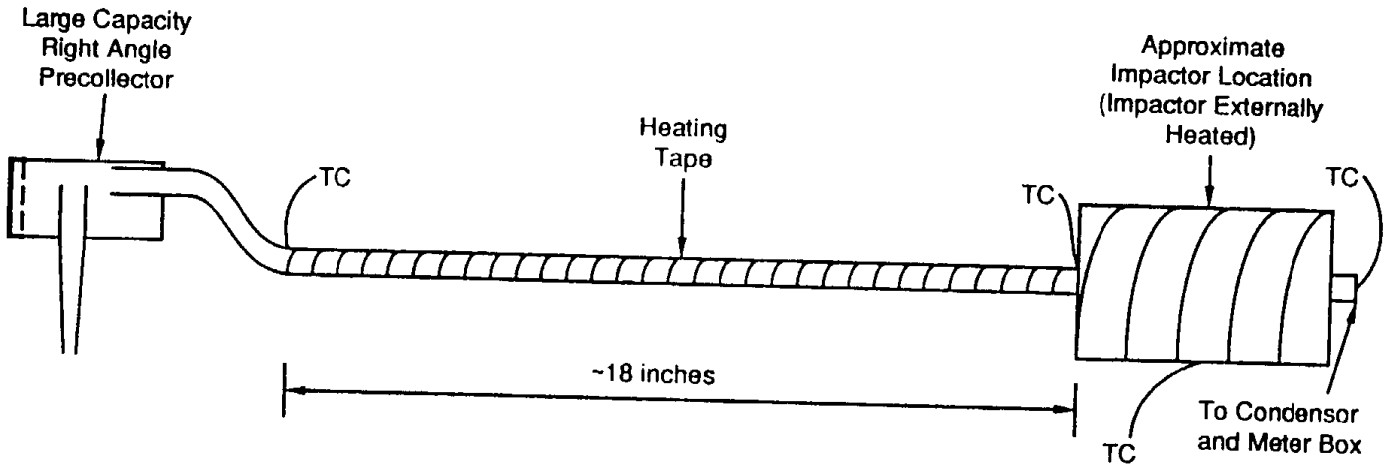
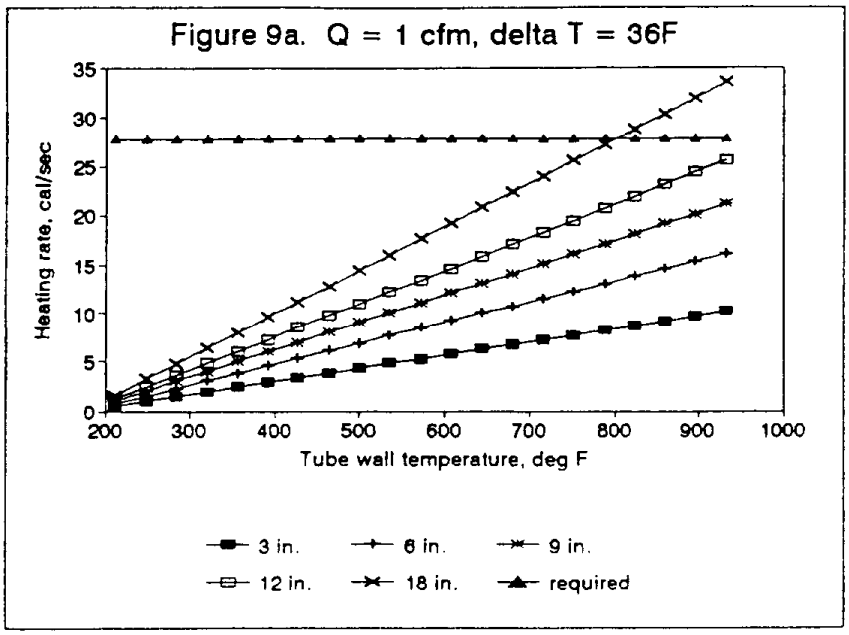


Figure 8. Layout of the sampling system for use in high moisture sources with condensed and/or entrained water present.



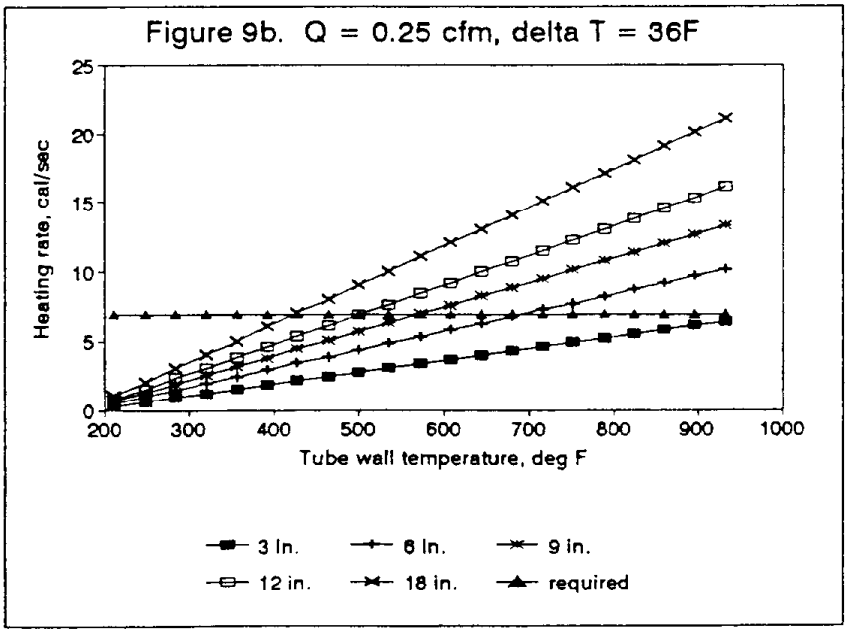


Figure 9. Heating rates delivered by various lengths of heated tubes for sample flow rates in the range of those normally used when sampling with inertial sizing devices.

Another factor that must be considered is whether there will be sufficient time for the largest droplets to be evaporated before exiting the heating tube and entering the inertial measurement device. Again using equations from Cooper (1976), the time required to evaporate a 20 μm droplet in a gas stream 36°F warmer than the droplet is shown as the top horizontal line in Figure 10 (about 0.2 sec). If the gas stream is 54°F (30°C) warmer than the droplet, then the lower horizontal line pertains (about 0.12 sec). The residence times of any droplet moving in a 1 cfm flow through 0.5 inch diameter tubes of the indicated lengths are represented by the sloping lines in Figure 10, with the upper line being that for a flow rate of 0.25 acfm and the lower for a flow rate of 1 acfm. Note that there is not quite enough time for a 20 μm droplet to completely evaporate in an 18 in. tube that is heating the gas to a 54°F differential at the higher of the two flow rates. Slowing the flow to 0.25 cfm will extend the residence time so that a 20 μm droplet would be evaporated at either temperature differential with an 18 inch long tube. And if the higher temperature is used, the droplets will have enough time to evaporate in tubes as short as about 7 in.

In fact, a liquid mass concentration as great as 10% of the total mass flow in a stack or duct (gas + particles + droplets) for droplets smaller than 20 μm is very unlikely to occur. Such high concentrations are typical of those encountered upstream of entrainment fig 10 separators and mist eliminators in scrubbers for all droplet sizes taken together, including those that would be removed by the precollector in the proposed method. The amount of liquid water associated with droplets smaller than 20 μm is much more likely to be 1% or less of the total mass flow of gas and particles in the duct. Consequently actual heating requirements to evaporate the droplets passed by the precollector can generally be expected to be much lower than those given above.

3.2.3 Selected Methodology for High Moisture sources

Based on the assumption that the sizes of the particles that would be obtained downwind from a source after drying downwind are the primary concern of this study, the recommended method of particle size measurement was to use an inertial precollector to remove the drops that are too large to transport and dry in a heated tube. The remaining smaller droplets are then to be evaporated to dryness in a heated tube connecting the precollector to a standard cascade impactor. The development of such a system and procedure was approved by the ARB and a prototype precollector/heated-inlet-tube setup was fabricated. The precollector, illustrated in Figure 11, is a modification of the right angle precollector now used in the ARB particle sizing method. The modification had two goals. The first was to raise the size cut from the roughly 12 μm value obtained with the current right angle precollector to something closer to 20 μm, and the second was to increase the liquid holding capacity beyond that provided by the current precollector. These goals were obtained in-so-far as possible, consistent with the constraint that the sampler had to be able to be inserted into the duct through a four inch diameter sampling port. As was the case for the high-temperature precollector, the Liquid Droplet Precollector design was based on that of the EPA/SRI right angle precollector. Again,

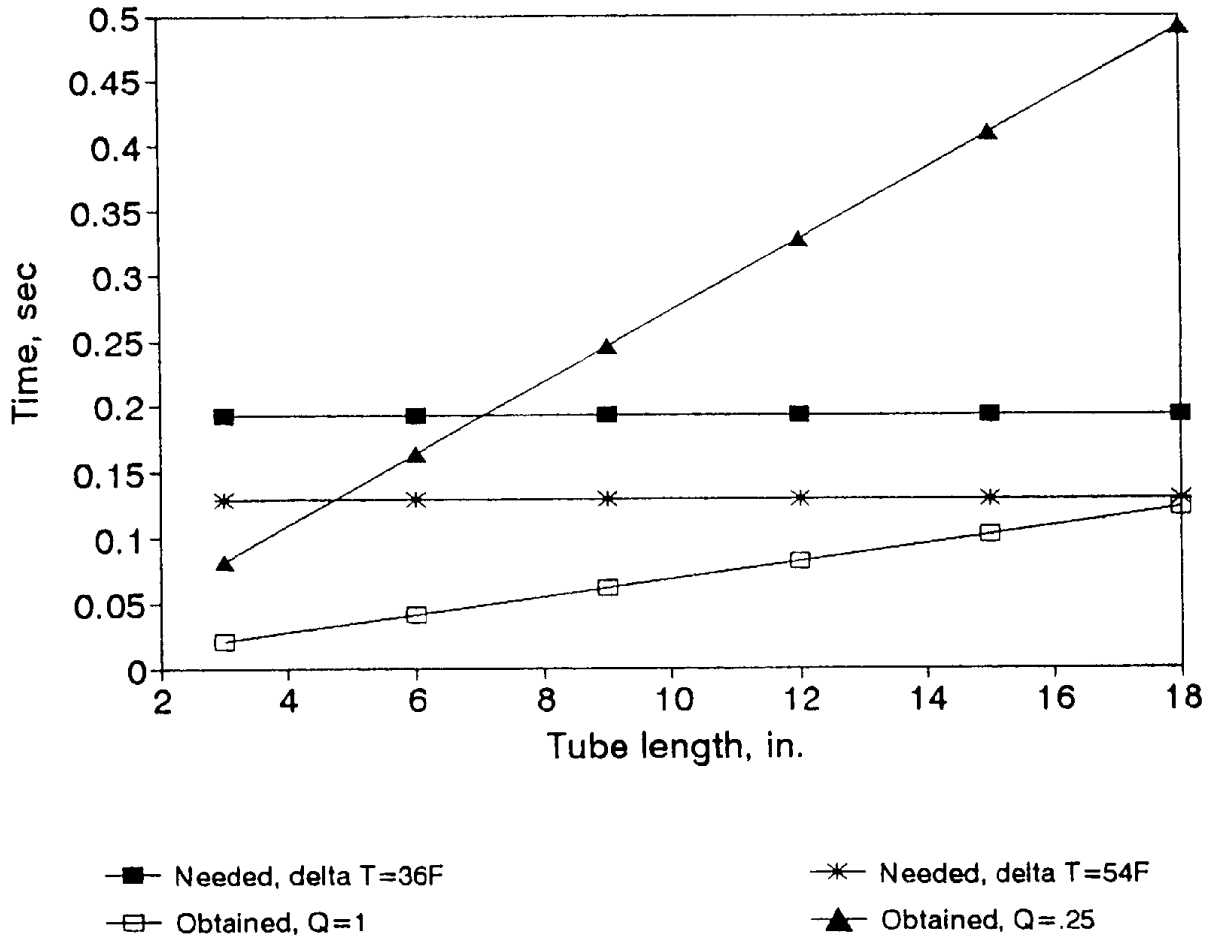


Figure 10. Residence times versus tube length for two sampling flow rates in the range of those used when sampling with inertial sizing devices.

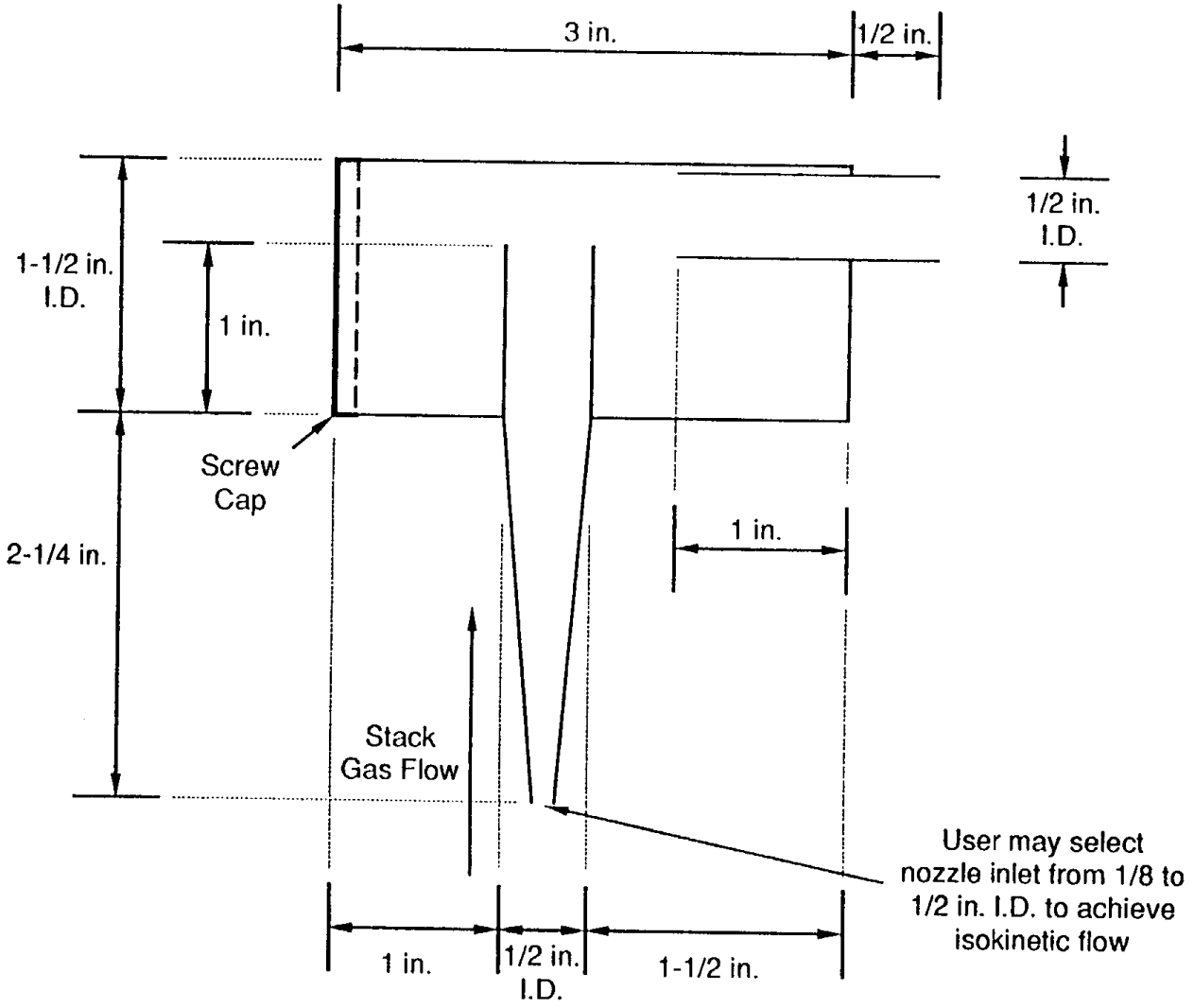


Figure 11. Design of the large droplet precollector for use in making particle size measurements in wet streams using inertial sizing devices.

calibrations for the SRI/EPA Right Angle Precollector are provided in Appendix C together with predicted values based on a semi-empirical theory for such devices developed at SRI. The ARB Liquid Droplet Precollector is based on the SRI/EPA design, but used longer nozzles to allow greater velocity dissipation and, consequently, permit larger precollector D_{50}s to be obtained. In addition, the volume of the precollector internal cavity was increased by lengthening it and the exit tube was positioned slightly further back in the cavity in order to increase the liquid holding capacity of the unit.

Detailed procedures for using the method are provided in Appendix B, "M501a - Particle Size Measurement for Wet sources." The proposed method provides data only for particles having dry residue diameters smaller than about 10 μm. If there is interest in identifying the droplet size distribution as well, then the KLD hot wire is recommended if drops larger than 250 μm are not present and the VDA is recommended if larger drops are entrained in the gas stream.

3.3 ARB Demonstration Tests

Demonstration tests were conducted at two sites to provide thorough shakedown tests of the suggested procedures and to familiarize ARB personnel with the hardware and software that were developed on the contract. These tests were conducted on March 14 and March 15 for the wet source method and March 17 through 19 for the hot source method. The sources used were a stack downstream of a scrubber used to control emissions from an incinerator for the wet source sampling demonstration and the exhaust of a landfill flare for the hot source demonstration. Three sampling runs were made at each source. Final revisions were made to the suggested methods based on the experience obtained during these demonstration tests.

The impaction substrates used in both test series were the quartz fiber type and quartz fiber backup filters were used as well. Preliminary weighing of the substrates was done at SRI's prior to shipment of the sampling equipment and materials. Prior to being used, the tare weights of several of the substrates were checked on the ARB laboratory balance and were found to be in satisfactory agreement with the SRI laboratory values. All final weighing was done at the ARB laboratory. A four-place (0.1 mg) balance was used for the demonstration tests because of the lack of availability of a five-place balance at the time the tests were conducted. This reduced the weighing precision somewhat, but had no real effects on the outcomes of the tests.

Upon conclusion of the tests, the prototype sampling hardware setups (precollectors and probes) were turned over to the ARB for its use in the future.

3.3.1 Wet Source Demonstration

The wet source method demonstration runs were made using single point sampling in a small diameter stack downstream of a scrubber used to control emissions from an incinerator. Two impactor configurations were used, each with six stages of separation with the LDPC providing a seventh stage. Two of the runs were made using plates 2, 3, 4, 5, 7, and 9 and one run was made using plates 3, 4, 5, 7, 9, and 11 of the UW V plate set.

The evaporator tube and the impactor were allowed one hour of preheat time prior to be being inserted into the stack and an additional 30 minutes of preheat was allowed after insertion into the stack to allow the precollector to reach stack temperature. The nozzle was covered with aluminum foil during the preheat periods. Because of the small diameter of the stack the impactor remained outside the stack throughout the operation. The evaporator tube and impactor were operated at nominal temperatures of 310°F and 270°F respectively for all three runs. Sampling times ranged from 35 minutes to one hour and were governed by the increase in operating pressure drop through the system as the sampling progressed during each run. The measured particulate concentration on the first test day was about 30 mg/dnm^3 and about 60 mg/dnm^3 on the second day of testing.

During the first run the stack temperature averaged about 158° and the moisture content was about 30%. During the second and third runs the stack temperature averaged about 166°F and the moisture content was about 35%. A few drops of liquid water was collected by the precollector during the first run, about 1.5 cc was removed by it during the second run, and a drop or so, at most, in the third run. After completing the third run, it was found that the nozzle orientation had been inadvertently confused and the nozzle had pointed downstream rather than upstream during the run. The latter may have contributed to the difference in the amounts of liquid removed by the precollector in runs two and three. The evaporative residues of the precollector catches were all quite small, ranging from 1 to 3 mg. No evidence was observed of any liquid being collected on the substrates within the impactor after any of the runs.

The bulk of the final dry particulate matter in each of the three runs was submicron in size with respectively 67%, 79%, and 75% in the fraction smaller than 1 μm. Most of the remainder (15% to 25%) was in the precollector catch. The D_{50} of the precollector as used during the demonstration runs was about 9.5 μm.

No operational difficulties were experienced during any of the runs that could be attributed to the equipment or procedures. Consequently, the method was deemed to have been demonstrated to perform satisfactorily. The weights of evaporative residues of the precollector catches and precollector and evaporator tube washes are provided in Appendix D. The net stage weights and reduced data from the three runs are also provided in Appendix D.

3.3.2 High Temperature Demonstration

The high temperature tests were conducted at the exhaust of a flare burning landfill generated gases. The gas temperatures as measured with unshielded, exposed-bead thermocouples ranged from 1650°F to 1840°F. The actual gas temperatures would have been about 150°F higher than the indicated values because the thermocouples, being unshielded, would have been cooled by radiation.

The same type impactor substrates and filters were used for these runs as were used for the wet source demonstration. Impaction substrates made from pre-baked Koawool paper were used in the precollector. Even though they had been prebaked, a control substrate used in addition to the real substrate during one run showed a weight loss of about 1.5mg that was attributable to the exposure to the high temperature gas stream. Consequently the suggested method has been amended to include a blank substrate in the precollector for each run.

The source for these tests was essentially a clean natural gas flame with entrained ambient air providing cooling gas and oxygen for combustion; consequently there was no expectation of actually being able to measure a particle size distribution for the source unless very long sampling times were used (on the order of days). Therefore a nominal sampling time of two hours was selected as being reasonable in terms of exposure to hot gas to identify operation problems without making the test day over-long. A nominal sampling rate of 0.4 dscfm was used for each of the three runs.

A warmup period of one hour was used to bring the probe internals and impactor up to near the target operational temperatures of about 400°F for the probe exit and 350°F for the impactor prior to insertion into the flare. After insertion, an additional 20 minutes of warmup time was used to allow the precollector and the hot end of the probe to equilibrate in temperature prior to the start of sampling. On the first day of testing some trash that had lodged in the cooling air hose when it was shipped restricted the flow and forced the use of a high blower power setting. After the problem was noted and the foreign material was removed very little of the blower capacity was needed to maintain the desired cooling of the probe internals. Temperature and power settings for the three tests are given in Tables 3.3, 3.4, and 3.5. The temperature of the sample tube inside the probe at a point a few inches back from the stack end of the probe ran about 850°F during these tests and could have been reduced further, had it been desirable, by increasing the cooling air flow.

After the probe was withdrawn from the stack at the end of each run the insulation and heating jacket was removed from impactor so it could cool and the cooling air was turned full on to cool the probe itself. A one hour cool-down period was found to be long enough to cool the probe sufficiently that a post test leak check could be performed, after which the impactor was removed, and the probe and impactor openings were covered with foil. They were then transported back to the motel for recovery and reloading as

TABLE 3.3

Probe Heater and Blower Power Settings and System Temperatures during ARB Demonstration Tests of the Prototype HT Particle Sizing Probe

HT Probe Run 1 3/17/94

					Gas Floor through Sampling System = = > >						
	Variacs, % of Maximum				Temperatures						
Time	Imp.	Probe Back	Probe Front	Blower	Stack	Probe 1	Probe 2	Probe 3	Probe 4	Tube	Impactor
1052	36	50	50	-	(80)	106	143	206			135
1115	36	50	50			181	317	378			190
1116	40	40	45								
1138	40	40	45		(84)	181	303	420			242
1139	44	40	45								
1205	44	40	45		(78)	179	299	410			302
1209 In Stack	44	40	45	0	1746	279	310	410			308
1214	44	40	45	0	1720	1000	650	394			316
1220	44	45	40	High, 100%	1675	779	438	309			316
1221	44	50	40	High, 100%							
1230 Start	44	50	40	High, 100%	1707	927	489	338			327
1242	46	50	40	"	1630	993	624	428			350
1242	41	50	30	"							
1250	40	50	30	"	1725	995	628	443			363
1254	32	50	Off	"							
1300	30	50	"	"	1717	992	621	442			358
1310	30	50	"	"	1742	1045	687	454			360
1320	30	40	"	"	1684	1040	684	423			365
1330	28	40	"	"	1725	1042	688	394			363
1340	28	40	"	"	1725	1024	676	395			361
1350	28	40	"	"	1775	1038	681	392			361
1400	28	40	"	"	1720	1031	687	399			361
1410	28	40	"	"	1700	1003	644	399			363
1420	28	40	"	"	1710	989	628	395			365
1430 End											

TABLE 3.4

Probe Heater and Blower Power Settings and System Temperatures during ARB Demonstration Tests of the Prototype HT Particle Sizing Probe

HT Probe Run 2 3/18/94

					Gas Floor through Sampling System = = > >						
	Variacs, % of Maximum				Temperatures						
Time	Imp.	Probe Back	Probe Front	Blower	Stack	Probe 1	Probe 2	Probe 3	Probe 4	Tube	Impactor
0825 Preheat	44	50	44	Off							
0830	44	50	44	"	67	80	126	118			196
0844	44	50	44	"	67						212
0957	44	50	44	"	80	183	290	531			355
0958 In Stack	44	50	Off	Low, 80%							
1016 Start	44	50	"	"	1700	770	609	352			361
1025	40	51	"	"	1680	864	762	423			369
1030	38	49	"	"	1690	807	754	432			378
1038	32	48	"	Low, 82%"	1680	846	736	426			376
1048	32	48	"	"	1660	842	725	425			383
1057	30	48	"	"	1680	838	721	422			385
1107	24	48	"	"	1720	866	743	424			380
1116	24	48	"	Low, 84%	1680	865	740	427			377
1126	24	48	"	"	1700	846	735	427			377
1138	24	48	"	"	1630	839	724	420			378
1148	22	48	"	"	1650	844	720	417			372
1154	22	48	"	"	1660	837	717	416			371
1206	22	48	"	"	1680	853	736	418			370
1216 End	22	48	"	"	1750	876	766	417			365

TABLE 3.5

Probe Heater and Blower Power Settings and System Temperatures during ARB Demonstration Tests of the Prototype HT Particle Sizing Probe

HT Probe Run 3 3/19/94

					Gas Floor through Sampling System = = > >						
	Variacs, % of Maximum				Temperatures						
Time	Imp.	Probe Back	Probe Front	Blower	Stack	Probe 1	Probe 2	Probe 3	Probe 4	Tube	Impactor
1008 In stack	46	42	30	Off	1625	163	165	468			283
1033	46	46	Off	Low, 65%	1700	796	562	358			327
1034 Start	46	46	"	"							
1044	40	46	"	"	1700	795	254*	391			339
1055	35	46	"	"	1680	795	209	410			347
1105	30	46	"	"	1640	810	241	414			350
1114	26	46	"	"	1670	758	201	411			349
1124	24	46	"	"	1720	799	203	414			347
1134	24	46	"	"	1700	806	233	415			349
1143	23	46	"	"	1690	810	227	416			350
1155	22	46	"	"	1770	849	236	421			349
1205	22	46	"	"	1780	856	240	425			348
1213	22	46	"	"	1840	834	243	424			350
1224	22	46	"	"	1700	831	265	423			350
1234 End	22	46	"	"	1780	769	238	418			351

* Thermocouple had detached from tube.

A blank or control wash-down of the probe and precollector was made after completing the recovery for the last run. Impactor substrate weight changes and the weights for the probe and precollector wash residues were all comparable to those for the blank stages and blank wash residues. Thus contamination of future samples by precollector and/or probe oxidation or disintegration products should not be expected to be a major problem. There was no visible evidence of damage to any part of the system other than discoloration from slight surface oxidation of the parts exposed to high temperatures. The weights of the precollector substrates and the dry residues of the precollector and probe washes are provided in Appendix D. The net stage weights and reduced data from the three runs are also provided in Appendix D.

Upon their conclusion the demonstration tests were deemed to have shown that the method and hardware performed satisfactorily.

SECTION 4

SOFTWARE

SRI provided the ARB with software for operating cascade impactors and reducing data obtained with them as part of a previous contract (ARB Contract A3-092-32, "Recommended Methodology for the Determination of Particle Size Distribution in Ducted Sources"). That software was written for the Apple II family of microcomputers. Updated versions of those impactor programs have been prepared for the ARB under the current contract. These updated programs are provided as executable files for use on IBM compatible PCs and are specific to the University of Washington (Pilat) cascade impactors, on which the current particle sizing method (ARB M501) and the new high temperature and wet source variants are based. The impactor hardware specific information from the previous Apple II programs has been updated to include the new special precollectors as first stage options. The EPA Method 201 PM10 cyclone and standard Method 5 sampling nozzles have also been added as options for use as the sampler entry/first-stage. The programs are written for general purpose application of the UW impactors and are not limited solely to high temperature or wet source applications.

There are four programs in the software package:

CARBD50X.EXE - Operational set-up program. Used to select jet plates, sampling system entry type (Precollector, cyclone, M5 nozzle...), nozzle tip size, etc., and sampling flow rate to be used. Input data needed are the bulk composition of the flue gas, the flue gas temperature, the gas velocity or pitot data, impactor temperature, barometric pressure, stack pressure, and the sampling system metering orifice constant. Outputs include the sampling flow rate for isokinetic sampling (at nozzle, impactor and standard conditions), the sampling rate to obtain the PM10 cut (if the PM10 cyclone is used as the entry), metering orifice pressure differential to obtain the desired sampling flow rate, and the stage cut diameters (D_{50}s) that would be obtained if the selected plates were used in the impactor.

CARBPRGX.EXE - UW impactor data analysis program. Used to reduce data from impactor sampling. Input data needed are the impactor train hardware set-up (entry type, nozzle tip size, and impactor stages used), bulk composition of the flue gas, the flue gas temperature, the gas velocity or pitot data, impactor temperature, barometric

pressure, stack pressure, the sampling system metering orifice constant and orifice pressure differential or the gas volume sampled from a dry gas meter and the gas meter calibration constant or the actual sampling flow rate, the duration of sampling, and the weights of the collected size fractions. Outputs include the sampling flow rate (at nozzle, impactor and standard conditions), the isokinetic ratio (as a percentage of isokinetic), the stage cut diameters (D_{50}s) that were obtained, and the size distribution in several formats (cumulative concentration, cumulative percentage, and concentration at size versus diameter).

STATIS.EXE - Program for averaging data from multiple runs. Permits outlier removal at the discretion of the user when three or more runs are averaged and provides standard deviations and confidence limits associated with the averages. The averaged results are provided in the same three formats used for the individual run results form CARBPRGX.

SYNTRAV.EXE - Program for synthesizing a traverse from several partial traverses. Intended for use in situations in which the velocity distribution is too skewed to permit a single impactor run to cover a full traverse without excessive isokinetic deviations.

Complete instructions for using the programs are provided in Appendix C, "Operational and Data Reduction Programs for University of Washington Cascade Impactors."

SECTION 5

SUMMARY

In summary, SRI recommends that modifications of the current ARB cascade impactor particle sizing methodology be used for sampling of both high temperature and high moisture gas streams. In each case large drops and/or particles are to be collected in adaptations of the currently used right angle precollector. In the wet stream sampling the remaining droplets are to be dried while passing through a heated inlet tube to the impactor. In the high temperature case the sampled gases are to be cooled to a temperature at which standard impactor hardware can be used by passing the sample through an air cooled tube after the removal of large particles by a special purpose precollector. These modifications of the present method have the advantages that (1) little in the way of additional hardware beyond that presently used will be needed, (2) little additional training of personnel will be required, (3) the data will be obtained on the same gravimetric basis as that being obtained currently, and (4) physical samples of the sized particles will be obtained for possible studies of composition and morphology. Prototype hardware for the application of the recommended methods was constructed, tested by actual use, and delivered to the ARB. Detailed procedures were written for the two measurement situations as variants to the existing ARB method for particle sizing, ARB M501 and computer software has been provided for operational set-up and data analysis using IBM compatible PCs.

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APPENDIX A

Method 501a
(Preliminary Draft)

Particle Size Measurement for Wet Sources

1 PRINCIPLE AND APPLICABILITY

This method is an adaptation/modification of the ARB Method 501, "DETERMINATION OF SIZE DISTRIBUTION OF PARTICULATE MATTER FROM STATIONARY SOURCES", that is intended to provide a means of measuring particle size distributions in sources containing entrained and/or condensed water droplets. General details of the method are as given in Method 501. This document covers the specific modifications to Method 501 that are needed for application of the method at "wet" sources.

1.1 PRINCIPLE

Particulate matter is withdrawn isokinetically from the source and segregated by size within in a cascade impactor that is operated *in situ*. The impactor is preceded by a precollector which removes the larger droplets from sample gas stream. The precollector is followed by a heated interconnecting tube in which any water on the particles passed by the precollector is evaporated. The sample stream then passes into the impactor, which is also heated to avoid recondensation of evaporated water, where the particles are collected in several size fractions for subsequent gravimetric or other analysis.

1.2 APPLICABILITY

This method is applicable in ducted sources with particulate mass concentrations in the range 0.005 to 50 grains per cubic foot, a pressure range of -20 to 20 inches water gage, moisture saturated gas streams with dew point temperatures from 32 to 180°F, and a velocity range of 5 to 60 feet per second. The method will provide size resolved particulate concentrations for all particles and the residues of droplets (after drying) for which the particles or the original droplets were smaller than the D_{50} of the precollector. It will also provide the total concentration of solids associated with particles and droplets present in the gas stream that were larger than the D_{50} of the precollector. The D_{50} of the precollector will typically be between 10 and 20 micrometers depending on the exact circumstances under which the sampling is carried out.

2 APPARATUS

The apparatus required for this method is the same as that required for Method 501 with additions described below. The method is predicated on the use of a University

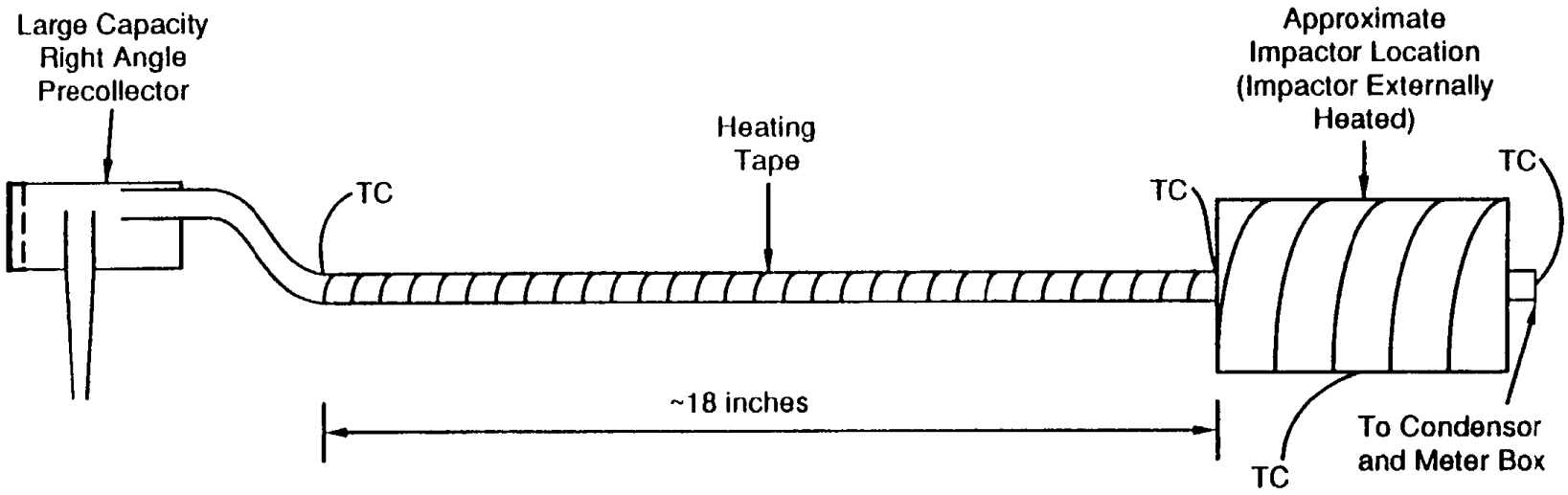

Figure 1. Recommended sampling system for ducted high-moisture emissions.

of Washington (Pilat) Mark V cascade impactor, but other impactors might be used in many instances. The basic setup of the precollector, heating tube, and impactor is illustrated in Figure 1. The items needed in addition to those called for in Method 501 are:

1. Liquid Droplet Precollector (LDPC). Custom design for the CARB - drawings are provided as an attachment to this method.

2. Heated interconnecting tube. 5/8 OD 316 SS, 20 to 24 inches long.

3. Heating jacket for the impactor body. Custom heating jacket from source provided in Section 2.3.

4. Thermocouples and thermocouple readouts for monitoring the wall temperatures of the heated tube and heating jacket for the impactor body.

5. Variable transformers or temperature controllers for the heated tube and impactor body heating jacket.

2.1 LIQUID DROPLET PRECOLLECTOR

The recommended liquid droplet precollector, or LDPC, is illustrated in Figure 2. This device is a modification of the EPA/SRI Right Angle Precollector (RAPC). It differs form the standard versions of the latter which are marketed by both Graseby/Andersen and Pollution Control Systems in the following respects:

The LDPC has a greater internal volume than the standard precollectors to permit collection and retention of relatively large quantities of water.

The exit tube of the LDPC has been moved off center toward the back wall of the device and lengthened to increase its liquid holding capacity in both the horizontal and vertical orientations.

The nozzle has been made a separate part from the impaction jet so that the jet can be permanently sealed to the precollector body to eliminate a possible source of leakage and consequent loss of sample.

And finally, the overall nozzle/jet length has been increased to provide a greater deceleration distance for droplets and particles, thereby making the cut size of the precollector as large as possible.

The LDPC and its nozzles are to be made from stainless steel to minimize corrosion and rust formation in and on the device. A standard commercial RAPC as described in the original Method 501 might be usable in instances where relatively small quantities of condensed/entrained water are present in the gas stream. However, the use

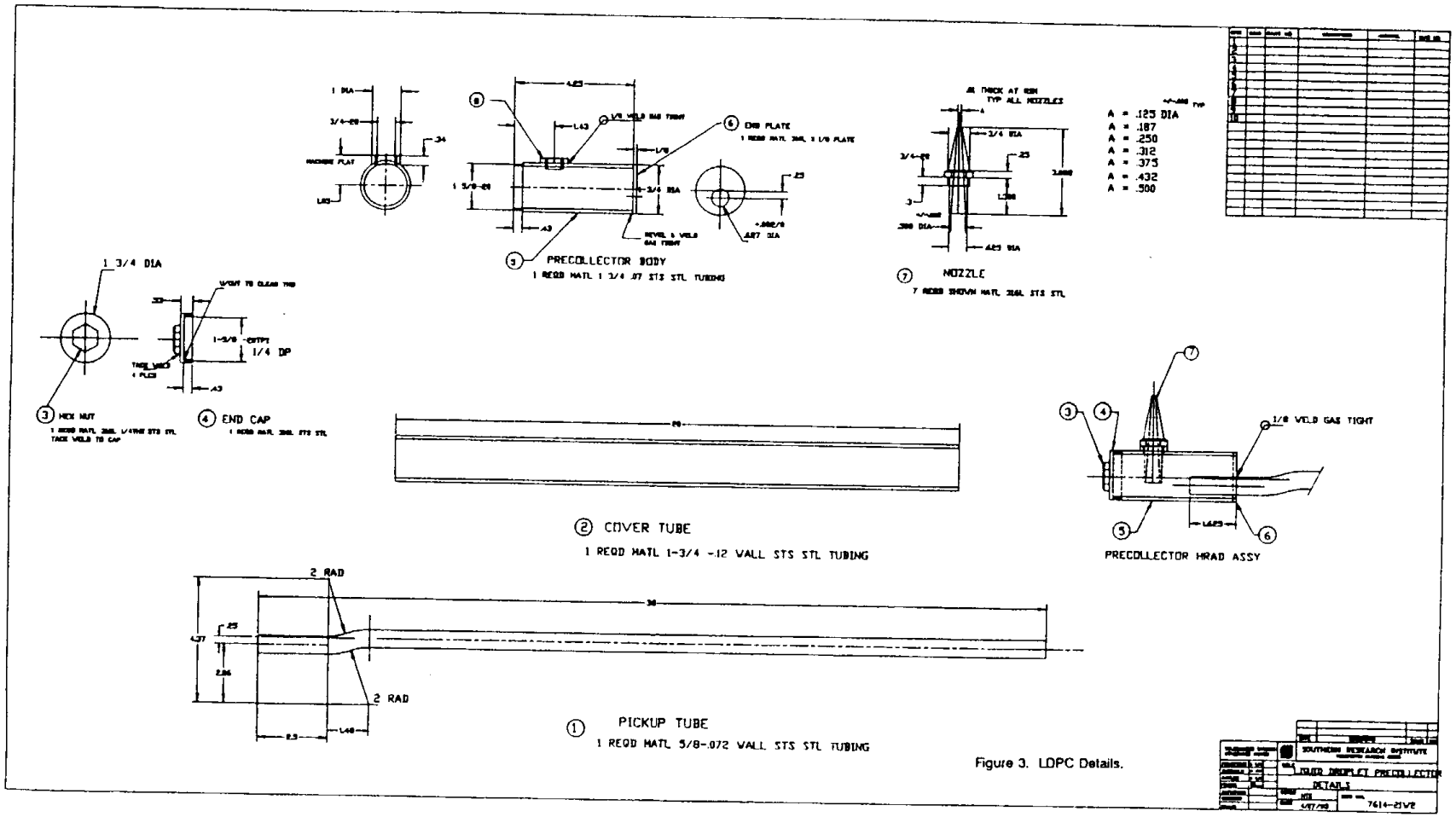

Figure 3. LDPC Details.

Figure 3. LDPC Details

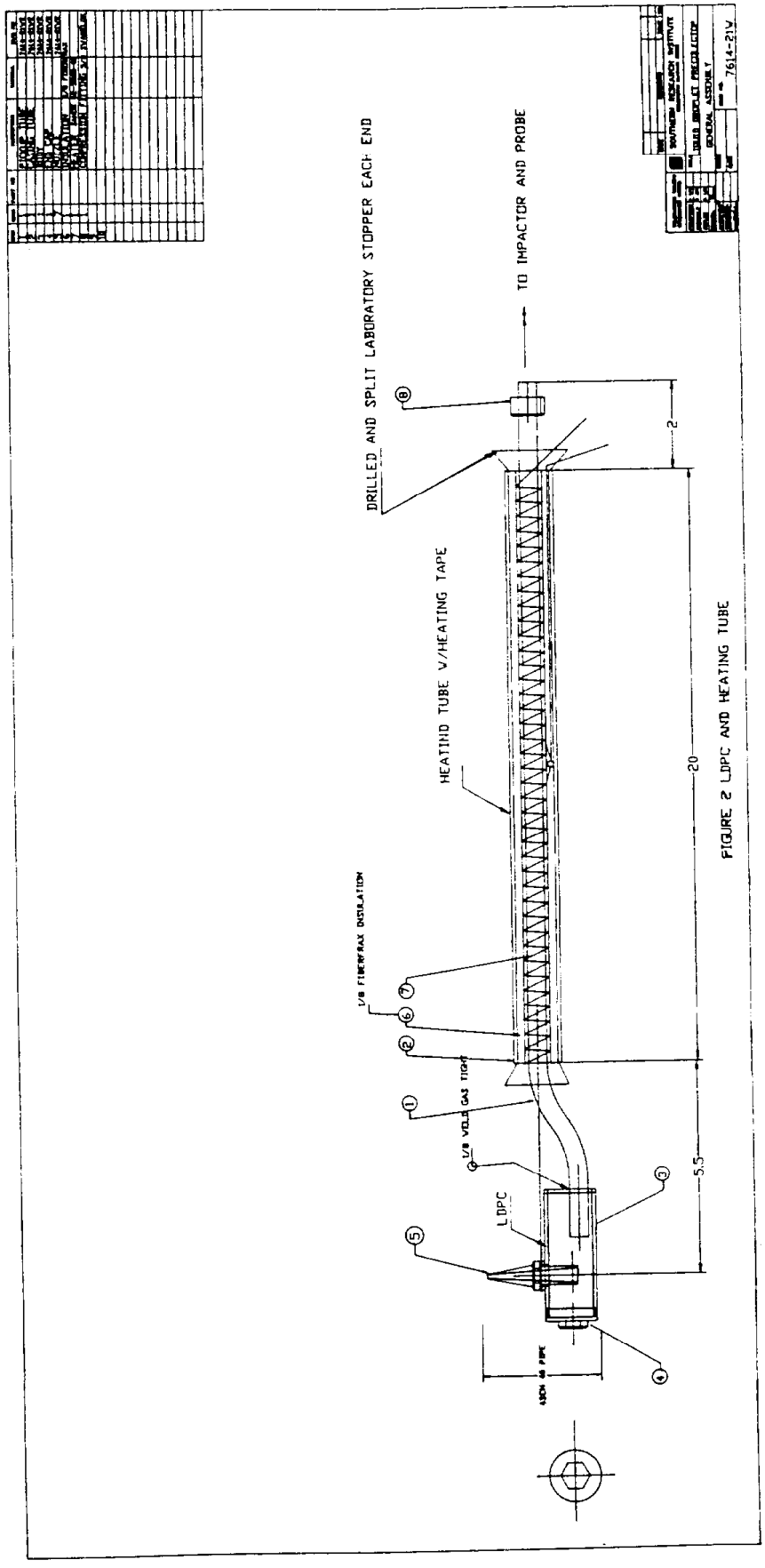

Figure 2. LDPC and Heating Tube

of the RAPC in a gas stream with a relatively high velocity would slightly reduce the range of resolved particle sizes from that which would be provided by the LDPC because of the shorter deceleration distance provided by the nozzles of the standard RAPC.

2.2 HEATED INTERCONNECTING TUBE

A heated interconnecting tube between the LDPC and the impactor is used to provide a zone for evaporating the droplets passed by the LDPC. A 0.5 inch id tube with a heated length of approximately 18 inches, heated to a wall temperature of 400°F, is required to reasonably insure that the water will be evaporated from any droplets passed by the LDPC. A connecting tube consisting of a 22 inch length of 5/8 OD, schedule 40, stainless steel tubing is recommended. The added length is needed to allow space for the attachment fittings at each end. A thermocouple should be mounted on the tube surface near the exit (impactor end) of the tube to permit monitoring of the wall temperature of the tube. A 48 inch long, approximately 200 watt, silicone rubber coated heating tape (Cole-Parmer Catalog No. G03111-40, 1/2 inch X 48 inch) is to be wrapped on the tube so that it spirals up the tube. Note: do not allow the tape to overlap on itself. The heating tape wrapped tube should be covered with a glass cloth insulating layer and an external stainless steel sheath as illustrated in Figure 3.

2.3 HEATING JACKET FOR THE IMPACTOR BODY

A custom silicone rubber heating jacket for the UW Mark III impactor shell is available from ElectroFlex Heat Inc., PO Box 88, Northwood Industrial Park, Bloomfield, CN 06002, as catalog number 867XXX. The jacket is sized and fitted with eyelets so that it simply wraps around the impactor and is secured by laces for use.

2.4 TEMPERATURE CONTROLS

Temperature control can be done either manually using a thermocouple readout switched between the two heater thermocouples to monitor the temperatures and variable transformers to set the applied voltages to control the temperatures or automatic temperature control devices compatible with the thermocouples and heaters can be used. The wall temperature of the heated tube should be 400°F (204°C) near the exit end of the tube. The impactor surface temperature should be set at 36°F (20°C) above the gas stream (saturation) temperature. The temperature limit of both the heating tape and heating jacket is 450°F (232°C). If the impactor probe geometry permits, the gas temperature at the exit of the impactor should be monitored and maintained at 36°F (20°C) above the flue gas temperature rather than relying on the wall/heater interface temperature as the controlling factor.

2.5 COLLECTION MEDIA

The use of fiber mat collection substrates for the impactor stages is recommended for this application. The latter have some capacity for retaining liquids without letting them flow off the surface should some liquid droplets reach the substrates - this is unlikely to happen but taking the precaution might be worthwhile. Either glass or quartz fiber substrates may be used but the quartz fiber type is preferred.

2.6 SAMPLING TRAIN AND OTHER APPARATUS

The remainder of the sampling train is the same as that for Method 501 and all other pieces of apparatus required are the same as those for Method 501 as well.

3 REAGENTS AND CONSUMABLES

The reagents and consumables required for this application are the same as those for Method 501.

4 PROCEDURES

4.1 GENERAL PROCEDURES

All procedures for the application of this method are the same as those for Method 501 other than the specific additions which follow.

4.2 WET SOURCE SPECIFIC PROCEDURES

4.2.1 SAMPLING TRAIN

The sampling train, except for the impactor, heating tube, and LDPC shall be assembled and leak checked as per Method 501. The impactor shall be loaded as described in Method 501 with preweighed collection substrates and filter together with a control substrate and control filter.

A thermocouple must be mounted on the wall of the heating tube near the exit end of the tube and the heating tape must be wrapped over the tube so that it passes over the thermocouple. Either a tubular insulating shroud must then be placed over the heating tube or the heating tube must be wrapped with glass cloth insulation. If a tubular shroud is used, end seals should be used to keep moisture from penetrating to the heater. These

can be made by drilling centered holes in laboratory rubber stoppers which are then slit along a radius from the center to the outside so they can be placed around the tube. By pressing them into the shroud ends the center hole and slit will be compressed, forming a water tight seal. Alternatively, if a glass cloth wrap is used as an insulator, the cloth can be wrapped with aluminum foil secured with glass electrical tape to keep it dry.

The precollector, heating tube, and impactor shall be assembled as a unit and the heating jacket shall be mounted on the impactor. The impactor/tube/LDPC assembly shall be mounted on the probe and the entire system shall be leak checked as per Method 501. Once the leak check has been passed, the impactor should be wrapped with glass cloth, the power leads and thermocouples should be hooked up, and the impactor body with its heating jacket and glass cloth wrapping should be wrapped with aluminum foil. The foil wrapping should be secured with glass electrical tape. The foil keeps the glass cloth insulation on the impactor from getting wet, thereby insuring that the heating jacket can perform properly. Alternatively a custom shroud can be used to keep the heater dry. Power should then be applied to the heaters, including the probe heater and all parts should be allowed to come up to the operating temperatures (450°F for the tube, 36°F above the stack gas temperature for the impactor shell, and 250°F for probe). A 1 hour warm up time should be given for the impactor internals to come to temperature once the shell has reached the desired temperature.

The areas of the connections at the inlet and outlet of the impactor should be insulated after the system is assembled and leak checked to minimize the possibility of condensation taking place in those areas.

4.2.2 SAMPLING

After the warmup is completed, sampling is done per Method 501. Data logging, *etc.* are as described in Method 501. Once sampling has begun and for all times thereafter until recovery of the contents of the precollector is complete, the orientation of the LDPC should be maintained as it was during the sampling. This should be done to avoid the possibility of spillage from the LDPC. In any case, either the nozzle or the exit end of the precollector must be the lowest portion of the precollector. It is essential that the heating system of the probe on which the LDPC/heating tube/impactor are mounted functions properly; otherwise condensation will take place in the probe which might drain back into the impactor, thereby invalidating the data from the run. A useful precaution to minimize the possibility of inadvertently allowing condensation to drian from the probe into the impactor is to operate the unit with the probe tilted slightly off the horizontal so that the impactor end of the probe is kept higher than the exit end.

4.2.3 RECOVERY

Upon completion of sampling, the LDPC/impactor assembly must be removed from the duct with the nozzle held pointing down at all times until the LDPC can be removed from the assembly. The LDPC can then be stood on its exit end, or it can be maintained in a horizontal position with the nozzle down. The nozzle and exit end should be covered with foil to minimize contamination and/or losses. The LDPC should then be taken immediately to a suitable location and its contents should be recovered. It is imperative that this be done quickly so as to minimize evaporative loss of the liquid contents. The impactor and heating tube can then be removed from the probe and taken to the recovery area.

4.2.3.1 LDPC RECOVERY

If auxiliary measurements of the concentration and size distribution of liquid droplets are being made in conjunction with the impactor runs (see note below), the quantity of liquid in the LDPC should be measured prior to evaporating its contents to determine the weight of the dry solid residue. After measuring the quantity of liquid in the LDPC, it should be rinsed with distilled water or acetone and the rinsate should be combined with the drained precollector liquid to be evaporated.

Note: Auxiliary measurements using the KLD AIMS hot wire device, the SRI Video Droplet Analyzer, the Insitec PCSV single particle light scattering probe, or other techniques might be used in conjunction with impactors to extend the range of size resolved data to diameters larger than the upper sizing cutoff of the impactor and precollector which will typically be about 10 μm. Any of the devices mentioned above can provide data on the size distribution of droplets in the gas stream within their individual operating size range limits. However, they do not provide any information on the concentration of dissolved and/or suspended solids within the drops, nor do they provide data on the final sizes of the droplet residues after the droplets are discharged from the stack and subsequent evaporation has taken place. The subject of dealing with such auxiliary data will be discussed in Section 6.

4.2.3.2 HEATING TUBE AND IMPACTOR RECOVERY

The interior of the heating tube should be brushed and rinsed and any particulate matter recovered should be considered a part of the catch of the first internal impactor stage. The remainder of the impactor should be recovered as per Method 501.

5 CALIBRATION

All calibration is as per Method 501.

6 DATA ANALYSIS

Data analysis is per Method 501. A group of computer programs is available through the ARB to facilitate data reduction. These programs are specific to the University of Washington (Pilat) Mark V cascade impactor in its various forms of application. Calculation of the D_{50} of the LDPC is included as an option in the program. The weight of the dry residue is to be used as the LDPC catch when analyzing the data. The size distribution generated will represent that which the emissions would present after drying in the plume downwind of the source for particle diameters smaller than the LDPC D_{50}. The extrapolation for diameters greater than the LDPC D_{50} will match the actual mass concentration; however, the detailed distribution may not represent the actual structure of the distribution very well. If detailed resolution is needed for the larger sizes, additional methods must be employed to obtain the drop size distribution before drying and assumptions must be made regarding the partitioning of the dissolved and suspended solids in the original droplets. In the simplest case, one might assume the solids to be homogeneously distributed in the droplets, in which case the size distribution of the dried aerosol would follow that of the parent droplets with a shift in diameters given by:

$$D_f = D_i * (Conc.)^{1/3}$$

where

D_f = Final Diameter
D_i = Initial Diameter

and Conc. = Solids concentration in the droplet.

The concentration associated with the diameter D_f is then the concentration of the entrained liquid in the gas stream at the diameter D_i multiplied by the mass fraction of solids in the droplets.

APPENDIX B

Method 501b
(Preliminary Draft)

Particle Size Measurement for High Temperature Sources

1 PRINCIPLE AND APPLICABILITY

This method is an adaptation/modification of the ARB Method 501, "DETERMINATION OF SIZE DISTRIBUTION OF PARTICULATE MATTER FROM STATIONARY SOURCES", that extends the method to cover measurements at sources that have gas temperatures in the range of 500°F to 2000°F. General details of the method are as given in Method 501 which covers applications at sources having temperatures below 500°F. This document covers the specific modifications to Method 501 that are needed for application of the method at high temperature sources.

1.1 PRINCIPLE

Particulate matter is withdrawn from the source as nearly as possible at isokinetic conditions, consistent with a fixed sampling rate, and segregated by size within a cascade impactor that is operated *ex situ*. The impactor is preceded by a precollector, operated *in situ*, which removes the larger particles from the sample gas stream. The precollector-impactor connection is made by means of a sheathed probe with the impactor mounted at the external end of the probe. Cooling air is forced through an annulus between the sample tube within the probe and the sheath at a rate such that the sample gas temperature is dropped to approximately 350°F before it enters the impactor. The sample stream then passes into the impactor, which is heated to 350°F to maintain the sample at that temperature while it passes through the impactor, where the particles are collected in several size fractions for subsequent gravimetric or other analysis.

1.2 APPLICABILITY

This method is applicable in ducted sources with particulate mass concentrations in the range 0.005 to 50 grains per cubic foot, a pressure range of -20 to 0.5 inches water gauge, a temperature range from 500°F to 2000°F, and a velocity range of 5 to 60 feet per second. The method will provide size resolved particulate concentrations for all particles smaller than the D_{50} of the precollector. It will also provide the total concentration of solids and condensibles which condense at temperatures of 300°F and higher that were present in the sampled gas stream. The D_{50} of the precollector will typically be between 10 and 20 micrometers depending on the exact circumstances under which the sampling is carried out. Sampling times will range from a few minutes to several hours, depending on the particulate concentration and size distribution encountered at a source. Because of relatively long heat-up before, and cool-down times after, sampling, the total time required

to take a single sample can be expected to be about the actual sampling time plus about 5 hours. Therefore only one or perhaps two samples can be obtained in a normal day in the field.

2 APPARATUS

The apparatus required for this method is the same as that required for Method 501 with additions described below. The method is predicated on the use of selected stages of a University of Washington (Pilat) Mark V cascade impactor, but other impactors might be used in many instances. The impactor is expected to be used in a "Mark III" configuration as described in Method 501, with the jet plates selected based on the sampling flow rate to be used. The latter will be based on the gas velocity in the duct, the available nozzle tip sizes, and the particulate concentration encountered. The basic setup of the precollector, probe, and impactor is illustrated in Figure 1. The items needed in addition to those called for in Method 501 are:

1. High Temperature Precollector or HTPC (Custom manufactured for the CARB - plans are available for duplication). Materials of construction are Hayes 230 and/or Hayes 556 high temperature alloys.

2. Sheathed air-cooled probe (Custom manufactured for the CARB - plans are available for duplication).

3. Heating jacket for the impactor body (ElectroFlex Heat Inc., PO Box 88, Northwood Industrial Park, Bloomfield, CN 06002, catalog number 867XXX) and high temperature (Samox) heating tapes for the rear of the sample gas tube (Cole-Parmer Catalog No.: G03115-40, 1/2 inch X 48 inch Samox Tape) and front of the sample tube (Cole-Parmer Catalog No.: G03115-20, 1/2 inch X 24 inch Samox Tape).

4. Thermocouples and thermocouple readout device for monitoring the wall temperatures of the flue gas, probe, and impactor body. Omega Type K thermocouples with XC (braided ceramic) insulation are recommended for the high temperature thermocouple applications.

5. Variable transformers or temperature controllers for the impactor and sample gas tube exit end heaters.

6. Blower (W.W. Grainger HP33 Cadillac Blower) and blower speed controller (Stacor Auto-Variable transformer (or Variac)) to provide the probe cooling air.

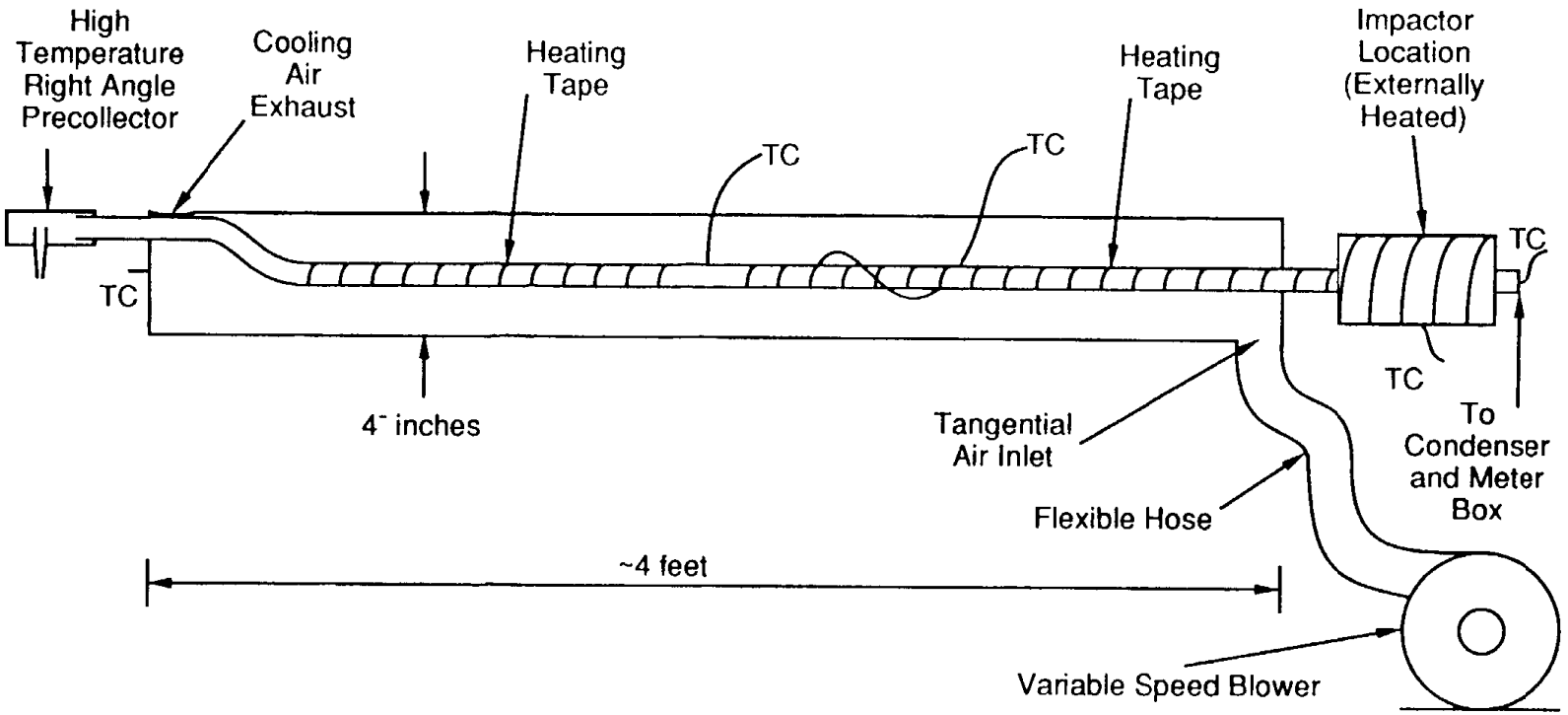

Figure 1. Prototype Sampling System for Ducted High-Temperature Emissions.

2.1 HIGH TEMPERATURE PRECOLLECTOR

The high temperature precollector, or HTPC, and its integral inter-connecting tube is illustrated in Figure 2. This device is a modification of the EPA/SRI Right Angle Precollector (RAPC) described in Method 501. It differs from the standard versions of the latter (which are marketed by Graseby/Andersen and Pollution Control Systems) in the following respects. The HTPC is constructed of Haynes 230, a "super-alloy" developed for use in high temperature regions of incinerators. Seals are either metal-to-metal or are provided by custom gaskets, developed by SRI for high temperature applications. Instructions for making and installing these gaskets are given in Section 4.2.1. The HTPC cap is secured by disposable bolts which pass through holes bored end-to-end through the precollector and the cap. This eliminates the use of threads on the cap which should reduce difficulties in disassembly due to galling of threads. [These nuts and bolts are also made of Haynes 230 (1/4 -20 by 3 1/2 inch long bolts and matching nuts, United Titanium, 3450 Old Airport Road, Wooster, OH 44691 (2160 264 2111)).] And finally, the overall nozzle/jet length has been increased to provide a greater deceleration distance for the sampled gas and particles, thereby making the cut size of the precollector as large as possible. The HTPC has a design temperature limit of 2000°F, but can probably be used successfully for limited exposures at somewhat higher temperatures and might withstand use to 2500°F (which is the suggested useful limit by the makers of the alloy).

2.2 SHEATHED PROBE

A 45 inch length of 0.625 od/0.5 inch id Haynes 556 tube is welded to the HTPC exit. This tube is passed through a 3.5 inch diameter sheath to a swage type connection at the impactor inlet. Cooling air provided by a blower is passed over the sample gas tube within the sheath from the external end of the probe and is exhausted through a hole in the downstream side of the sheath at the HTPC end of the probe. The cooling air is used to reduce the gas temperature of the sample to about 400°F before it enters the impactor and helps protect the probe by cooling it as well. A high temperature heating tape is wrapped on a portion of the gas tube within the probe near the exit end to keep excessive cooling of the part of the tube outside the duct from taking place. A second high temperature heating tape is mounted within the probe on the sample tube at the HTPC end of the probe to provide a means of preheating the tube prior to insertion into the duct. Thermocouples should be mounted on the tube surface near the exit (impactor end) of the tube and at other locations along the tube to permit monitoring of the wall temperature of the tube. The probe is illustrated in Figure 3. A portion of the heating tape used for preheating the part of the probe external to the stack may be exposed to high temperatures at times. Consequently the heating tape must be capable of withstanding high temperatures. The SAMOX® type heating tapes, which will withstand temperatures to 1400°F, are satisfactory for this purpose.

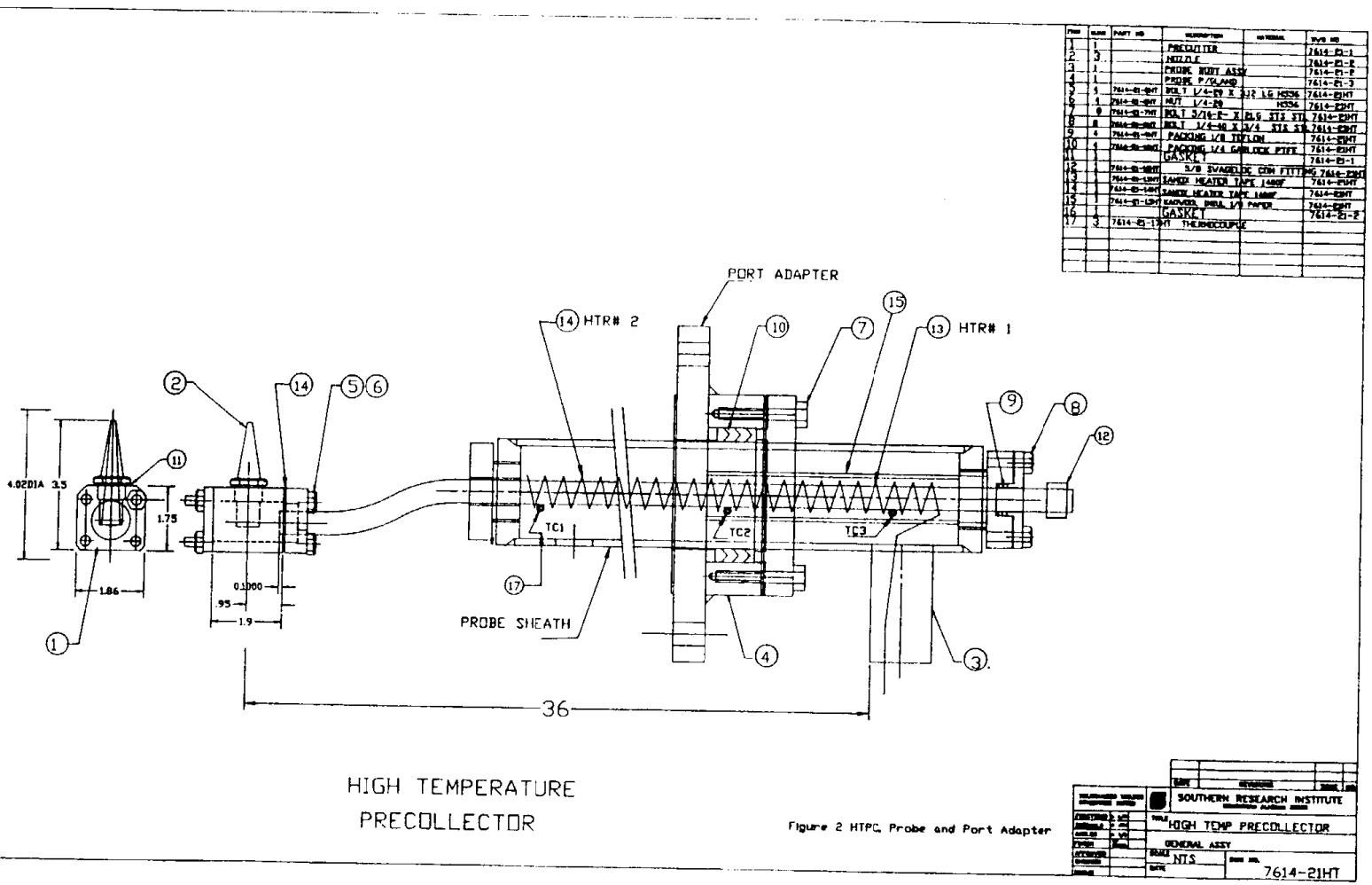

Figure 2. HTPC, Probe and Port Adapter

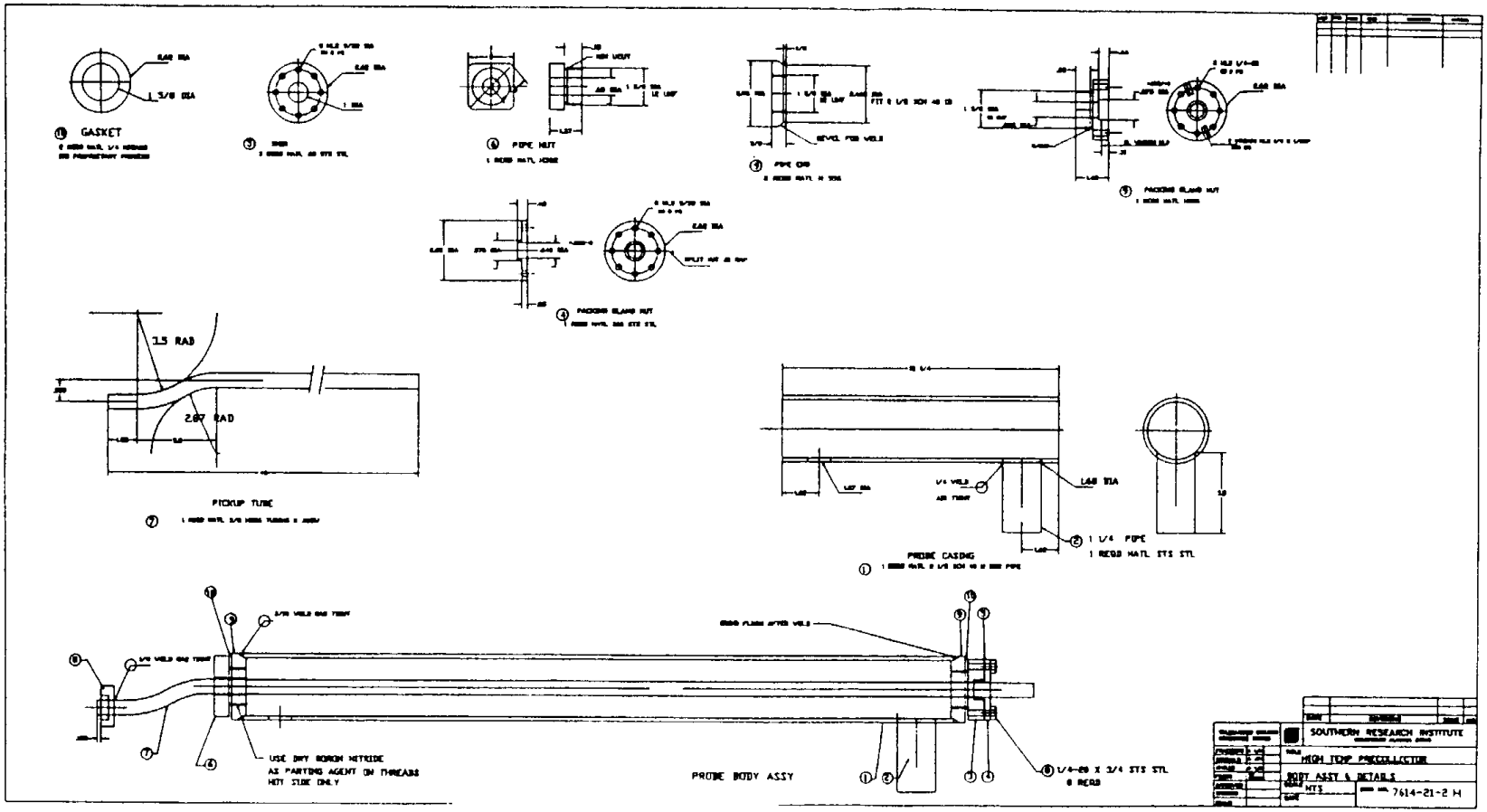

Figure 3. HT Probe Details

2.3 HEATING JACKET FOR THE IMPACTOR BODY

A custom silicone rubber heating jacket for the UW Mark III impactor shell is available from ElectroFlex Heat Inc., PO Box 88, Northwood Industrial Park, Bloomfield, CN 06002, as catalog number 867XXX. It simply wraps around the impactor and is secured by laces for use. The temperature limit for the heating jacket is 450°F.

2.4 TEMPERATURE CONTROLS

Temperature control of the impactor body can be done either manually using a thermocouple readout and a variable transformer to set the applied voltage to control the temperature; or an automatic temperature control device compatible with the thermocouples can be used. The wall temperature of the portion of the gas tube within the stack is controlled by adjusting the flow rate of the cooling air by changing the blower speed. The wall temperature of the portion of the gas tube that is external to the stack is controlled by means of the heating tape. The wall temperature at the exit end of the gas tube should be in the range of 300°F (160°C) to 400°F (204°C). The impactor shell temperature should also be in the range of 300°F (160°C) to 400°F (204°C), with a target of 350°F and should not be allowed to exceed 400°F to protect the heating jacket, which has a temperature limit of 450°F.

2.5 COLLECTION MEDIA

The use of quartz fiber mat collection substrates for the impactor stages is recommended for this application. A rectangular piece of Pallflex QAST 2500 quartz fiber filter material can be used as a collection substrate in the HTPC for sample stream gas temperatures below 1500°F. At higher temperatures a piece of Kaowool® Felt or Ultrafelt Papers might be used. The latter products have temperature limits of 2300°F or higher. The Kaowool® Felts and papers are made with an organic binder that must be burned off before use. When these must be used, they must be pre-baked in an oven at a temperature that is high enough to burn out the binder or erroneous precollector sample weights will be obtained. An oven temperature of at least 500°F should be used and a much higher temperature is recommended if a suitable oven or muffle furnace is available. Alternatively, Haynes 230 or similar shim material might be used, or impaction might be allowed to take place directly on the wall of the precollector. The latter is not recommended because it is possible that some particles might be molten when sampled and might adhere more or less permanently to the precollector wall. The manufacturer's specifications for the Kaowool® products are provided in an appendix to this document.

2.6 SAMPLING TRAIN AND OTHER APPARATUS

The remainder of the sampling train is the same as that for Method 501 and all other pieces of apparatus required are the same as those for Method 501 as well.

3 REAGENTS AND CONSUMABLES

The reagents and consumables required for this application are the same as those for Method 501 with the following additions:

1. "M board" insulation (0.125" thick) from Thermal Ceramics (2102 Old Savannah Road, P.O. Box 923, Augusta, GA 30903).

2. Kaowool rigidizer (also obtained through Thermal Ceramics).

3. Boron nitride (BN)

4 PROCEDURES

4.1 GENERAL

All procedures for the application of this method are the same as those for Method 501 other than the specific additions which follow. Method 501 can be used with quartz fiber substrates and filters for applications at sources with gas temperatures below 800°F. Viton rubber o-rings should be used in the impactor under such conditions. The o-rings may harden when used and replacement of all of the o-rings in the impactor may be required for each run.

4.2 HIGH TEMPERATURE SOURCE SPECIFIC PROCEDURES

4.2.1 SAMPLING TRAIN

The sampling train, except for the impactor, sheathed probe, and HTPC shall be assembled and leak checked as per Method 501. The impactor shall be loaded as described in Method 501 with pre-weighed collection substrates and filter together with a control substrate and control filter. The precollector, probe, port adapter, and impactor shall be assembled as a unit and the heating jacket shall be mounted on the impactor.

The gaskets used for sealing the HTPC body/nozzle connection (and for providing some protection to the threads) must be made by the user. Obtain M board insulation (0.125" thick) from Thermal Ceramics (2102 Old Savannah Road, P.O. Box 923, Augusta, GA 30903). Cut the desired shape piece to use as a gasket. Compress the piece evenly in a laboratory press or using two pieces of steel plate and C-clamps to compress the 0.125" board to about 60% of its original thickness. Remove the compressed gasket and soak it completely in Kaowool rigidizer (also obtained through Thermal Ceramics). Prepare a

paste from Kaowool rigidizer and boron nitride (BN) (the resulting paste should have a consistency between jelly and toothpaste). Paint or coat the sealing surfaces of the still-wet compressed gasket with the paste, install the gasket, and tighten the connection. Bake or allow the assembly to air dry. This process has been demonstrated to create reasonably airtight seals that withstand temperatures up to 2000°F and the materials will withstand much higher temperatures.

It has been proven possible to obtain adequate seals between the precollector body and its base using just a metal-to-metal seal and the latter is recommended so as to minimize contamination of the sample. Kaowool paper can optionally be used to provide the seal between the nozzle and the HTPC body. A gasket should be used between the nozzle and body to protect the threads from being fouled by particulate matter.

All threaded surfaces that are in the high temperature gas stream must be coated with boron-nitride (BN). The coating acts as a parting and release compound to keep the connections from galling and seizing. This can best be done by making a thin water-based slurry of BN into which the parts are dipped or which is then painted onto the parts.

The tube portion of the stack end packing gland for the HTPC/impactor connecting tube must be slipped over the tube and slid up to the HTPC body before the tube is inserted into the sheath. In normal operation the precollector will project about one foot from the duct end of the sheath, leaving just enough of the tube projecting from the impactor end to allow the connection to the impactor to be made. Thermocouples must be mounted about four inches from each end of the connecting tube between the HTPC and impactor and other intermediate points as desired. These must be covered with high temperature insulating material (Kaowool, Kaowool paper, or similar materials) to insure that they indicate the tube wall temperature and are not influenced directly by radiation from the shroud or by contact with the heater for the impactor end of the tube. Kaowool paper secured by small SS hose clamps has been found to work well. The Samox heating tape should then be wrapped over that part of the connecting tube that will be outside the stack and covered with Kaowool paper to keep the sheath cooling air from overcooling the outer portion of the sample tube. A thermocouple should be placed on the tube under the heating tape near the exit end of the tube. This assembly must be slipped into the shroud through the shroud portion of the packing gland at the stack end of the probe after mounting the port adapter on the shroud. The thickness of the insulation over the tube heater must be governed accordingly. The tube heater leads and thermocouple leads must be fed through the cooling air inlet sidearm as the tube is fed into the shroud. Finally, the impactor end packing gland should be assembled and both packing glands should be partially tightened. The stack end seal gasket should be soaked in rigidizer before being mounted or it's usable life will be short.

The impactor shall be mounted on the probe/HTPC assembly and the entire system shall be leak checked as per Method 501. Teflon ferrules MUST be used for making the swage connection between the probe and impactor. The use of metallic ferrules will result in the capture of the sample tube in the sheath and subsequent removal of the tube from the sheath would be impossible without cutting the end of the tube off. These Teflon ferrules must be replaced with new ones for each sampling run. If the gaskets are omitted at the precollector, the leak check should be done with the precollector body/nozzle assembly removed from the precollector base. Once the leak check has been passed, the heating jacket and an insulating shroud should be mounted on the impactor, the power leads and thermocouples should be hooked up, and the blower hose should be attached. Cable ties provide a quick and easy means of securing the insulating shroud over the impactor and the exit end of the air-cooled shroud. The blower hose has a slot cut in the cuff that fits over the air inlet arm of the probe sheath. The heater and thermocouple leads should be fed through the slot and the cuff should be clamped on the inlet arm with a hose clamp, leaving as small a leakage path out of the slot as possible.

A 90° elbow and tube should be mounted at the impactor exit such that the tube will point down when sampling is in progress. This will insure that if any condensation takes place in the sample line following the impactor, it will not drain into the impactor where it might damage or contaminate the filter and substrates, resulting in the invalidation of the run.

The HTPC/tube/impactor assembly should then be rotated until the nozzle is pointing in some convenient and identifiable direction with respect to the cooling air inlet tube and/or the impactor exit tube. The orientation of the latter can then be used to align the nozzle into the flow for sampling when the probe is in the stack. In operation, the cooling air exhaust must point downstream when sampling is taking place to avoid the possibility of having cooling air dilute or contaminate the sample.

Power should then be applied to the heaters, including the sample gas tube exit heater. If Variacs are used, power settings of about 40% have been found satisfactory for warm-up and about 35% for sampling. **Full 110 volt power must not be applied to the heaters** - it will result in severe overheating and the destruction of the heaters, especially the silicone rubber coated impactor heater. The impactor should be allowed to preheat for about 45 minutes, after which the probe and HTPC should be inserted into the stack with the nozzle pointing downstream and all parts should be allowed to come up to the operating temperatures (300°F to 400°F for the gas tube exit end in the probe and the impactor shell temperature). A one hour warm-up time should be adequate for the impactor internals to come to temperature once the shell has reached the desired temperature. The internal temperature of the HTPC and the inserted portion of the probe will probably come to temperature within about 30 minutes after insertion. Once the thermocouples indicate that the temperatures have stabilized at the desired or acceptable values the probe can be rotated to point the nozzle into the gas flow and sampling can begin.

4.2.2 INSERTION AND REMOVAL

Although the probe/impactor assembly is light enough to be handled and carried by one person when cold, it is advisable that two people work together to handle it. This is especially recommended when it is fully assembled and even more so when it is hot. While it is warming up prior to being inserted into the duct or flue, the surface temperatures will probably be low enough that the chances of being burned are low, none-the-less, it is advisable that long-sleeved, natural fiber clothing be worn and that gloves be used. When removing the assembly from the port after sampling has been completed, it is imperative that protective clothing and "high-temperature" gloves be used in handling and working around the probe as the HTPC and outer end of the shroud will be at the flue gas temperature when the probe is removed from the duct or stack.

The safest way to handle the assembly for insertion and removal is to suspend it by **chains** (**not ropes!**) from two trolleys in a manner similar to that used for hanging Method 5 sampling trains. Ropes and a pulley system can be used on the trolley, environmental conditions in the working area permitting, to allow height adjustment and provide a means of raising and lowering the probe from/to the floor, but the ropes should be attached to chains which, in turn, are attached to the probe assembly and not directly to the probe. If ropes are used, they should be made of natural fibers as the latter will tolerate contact with high temperature surfaces better than nylon or other artificial fibers commonly used in ropes. One such adjustable height suspension point at each end of the assembly will allow the probe to be positioned, inserted, and removed without the necessity of using a lot of strong, long-duration manual contact with it. This is of paramount importance when removing the probe after sampling when it is a safety hazard because of its high temperature surfaces. If the port adapter is used as one of the attachment points, the assembly can be fairly easily guided to the port and aligned for insertion and then, later, can be fairly easily removed without a lot of gripping and direct handling of hot masses of metal. Although two people can manage insertion and removal, it is far easier for three people to do the job - one each on the two suspension hangers to adjust height and level and the third to guide the probe into/out-of the port and attach/release the port adapter.

4.2.3 SAMPLING

After the warmup is completed, sampling is done per Method 501. When sampling commences the sample-line heater power will probably have to be reduced and the cooling air flow will have to be brought up to obtain and hold the desired impactor inlet gas temperature. Data logging, *etc.* are as described in Method 501. The sample line thermocouple readings should be recorded in addition to the data called for Method 501.

It is essential that the probe heating/cooling system function properly; otherwise condensation may be take place at any low temperature zones; and damage to the probe and impactor might result from excessively high temperatures. The size cuts produced by the impactor stages are only slightly sensitive to the gas temperature within the impactor; therefore the exact temperature of the gas as it passes through the impactor is not required and can be taken to be the temperature of the connecting tube exit wall and the impactor body, normally about 350°F.

4.2.4 SAMPLE RECOVERY

Upon completion of sampling, the HTPC/probe assembly must be removed from the duct and the entire assembly must be allowed to cool. Unless the ambient air is exceedingly dirty as compared to the stack gas, it is a good practice to pull one to two cubic feet of ambient air through the assembly immediately after removing it from the duct. This flushes the residual moisture that was brought in with the sample gas from the assembly, thereby reducing the chance of the back-up filter sticking to the filter holder assembly and it reduces the time required for the stage weights to equilibrate for weighing after being unloaded, as well. The blower can also be left on at a moderate flow rate to cool the probe more rapidly. The nozzle should be covered with foil as soon as it cools enough to permit foil to placed on it so as to minimize contamination and/or loss of sample during the cool down period. A cool-down time of about one hour will probably be adequate.

4.2.4.1 HTPC RECOVERY

After an adequate cool-down period (to avoid personal injury), the HTPC nozzle should be covered and the HTPC/probe/impactor assembly should be removed to a suitable recovery area. At the recovery area the impactor should be removed from the probe . The sheath may or may not have to be taken off the sample tube. If the probe can be successfully managed for recovery of any material deposited in it with the sheath in place, leaving it on is preferable as that minimizes turn-around time and possible damage to the thermocouples and sample-line heater and their leads.

At this point, the end cap and nozzle should be removed from the precollector, after which the precollector substrate can be removed. The precollector substrate should then be placed on a weighed piece of aluminum foil or one with which it was initially weighed. It is advisable to perform this operation over a piece of aluminum foil to prevent the loss of sample should any spillage occur. Care must be taken when breaking the gasket seals on the HTPC, if gaskets were used, to avoid letting any gasket material contaminate the sample. Any particulate matter deposited in the nozzle or that is loose in the precollector body should be brushed and/or rinsed with acetone onto the precollector substrate. The substrate should then be dried, first covered in a hood if an acetone rinse was used and then in a desiccator for moisture removal.

4.2.4.2 PROBE AND IMPACTOR RECOVERY

The sample gas tube should be cleaned in the same fashion as a Method 5 particulate probe. The residue from the probe should be inspected to insure that it is not dominated by corrosion of spallated material from the probe walls (not expected to occur) and, if found to be free of such material, should be dried and added to the catch of the impactor inlet stage. The remainder of the impactor should be recovered as per Method 501.

5 CALIBRATION

All calibration is as per Method 501.

6 DATA ANALYSIS

Data analysis is per Method 501. A group of computer programs is available through the ARB to facilitate data reduction. These programs are specific to the University of Washington (Pilat) Mark V cascade impactor in its various forms of application. Calculation of the D_{50} of the HTPC is included in the program. The program will provide an extrapolation of the particle size distribution for diameters greater than the HTPC D_{50}. The extrapolation will result in a match with the actual mass concentration; however, the detailed distribution in the extrapolation range may not represent the actual structure of the distribution. If detailed resolution is needed for sizes larger than the HTPC D_{50}, additional methods must be employed to obtain the size distribution for the larger sizes. This might be done by means of an *in-situ* device such as the Insitec PCSV single particle light scattering probe for sizes in the 5 to 50 μm range. Or the size distribution of the HTPC catch might be measured microscopically or instrumentally by such devices as the Sedigraph or Coulter Counter.